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Distribution of Heavy Hydrogen and Oxygen Isotopes in Water Distillation

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Abstract—The possibility of high-precision determination of mass transfer characteristics of stainless steel packings in the separation of hydrogen and oxygen isotopes by vacuum distillation of water in columns with a diameter of 60–250 mm was shown. An algorithm for calculating the parameters of separation and the concentration profile of deuterium and oxygen-18 during their simultaneous separation in a closed type unit with product selection by vacuum distillation of water was presented.

Keywords: deuterium, oxygen-18, water distillation

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INTRODUCTION

Vacuum distillation of water is one of the most widespread methods for the separation of hydrogen and oxygen isotopes [1]. Despite the low values of the single separation effect, the indisputable advantages of the method are the simplicity of apparatuses; no toxicity, corrosion, and explosion problems; and unlimited stocks of raw materials. As a result, in the mid-20th century, water distillation was used to solve the problem of obtaining heavy water (D_2O) from natural raw materials and then at the stage of final concentration of deuterium in the processing of substandard heavy water [1–4]. Water distillation is the only industrial method for the production of heavy-oxygen water $\left(H_2^{18}O\right)$ and deuterium-depleted water (DDW) with reduced deuterium (heavy hydrogen isotope) content, which are used in medical and biological research [5–7].

The isotope separation by distillation is based on the phase equilibrium between the liquid and the vapor. The isotope exchange of hydrogen and oxygen is described by Eqs. (1) and (2), with heavy isotopes concentrated in the liquid phase [1, 3]:

$$HDO_{(v)} + H_2O_{(l)} \Leftrightarrow H_2O_{(v)} + HDO_{(l)}, \qquad (1)$$

$$H_2^{18}O_{(v)} + H_2^{16}O_{(l)} \Leftrightarrow H_2^{16}O_{(v)} + H_2^{18}O_{(l)}. \qquad (2)$$

The thermodynamic equilibrium in these systems is characterized by a separation factor α equal to the ratio of the relative concentrations of the heavy isotope in the enriched and depleted phases:

$$\alpha = \frac{x(1-y)}{(1-x)y}. \qquad (3)$$

In this equation as applied to distillation, the variable x is the concentration (atomic fraction) of the heavy isotope (D or ^{18}O) for liquid water, and y is the same for vapor.

For binary mixtures, the separation factors can be calculated using the partial pressures of the isotope modifications of water molecules and empirical equations that are valid in the temperature range 273–403 K [1, 8]:

$$\alpha_{H-D} = \frac{P^0_{H_2O}}{P^0_{HDO}} = \sqrt{\frac{P^0_{H_2O}}{P^0_{D_2O}}}, \qquad (4)$$

$$\alpha_{H_2^{16}O-H_2^{18}O} = \frac{P^0_{H_2^{16}O}}{P^0_{H_2^{18}O}}, \qquad (5)$$

$$\ln\frac{P^0_{H_2O}}{P^0_{D_2O}} = \frac{26398.8}{T^2} - \frac{89.6065}{T} + 0.075802, \qquad (6)$$

$$\ln\alpha_{^{16}O-^{18}O} = \frac{1037}{T^2} - \frac{0.4156}{T} - 0.00207. \qquad (7)$$

The analysis of the separation factors for the H_2O–D_2O and $H_2^{16}O$ – $H_2^{18}O$ isotope systems given in Table 1 shows that the single separation effect for oxygen is substantially smaller than for hydrogen. As the temperature increases, α decreases, as a result of which the

Table 1. Temperature dependence of the separation factor during water distillation [1]

Isotope system	323 K	333 K	343 K	353 K	363 K
H_2O–D_2O	1.053	1.047	1.041	1.035	1.030
$H_2^{16}O - H_2^{18}O$	1.0078	1.0068	1.0063	1.0058	1.0053

process is performed in vacuum. In this case, the optimum pressure is at a level of 0.02–0.03 MPa, which corresponds to the temperatures of 60–70°C [1, 3].

The low enrichment factor ($\varepsilon = \alpha - 1$) in water distillation, especially in the separation of oxygen isotopes, necessitates the creation of countercurrent separation apparatuses with a large number of theoretical plates. The isotope mixtures to be separated are evidently H_2O–HDO and $H_2^{16}O - H_2^{18}O$. As the calculations of the separation apparatuses are usually performed using the general theory of fine separation, which takes into account only one isotopic system, it is interesting to determine the tendencies of variation of the deuterium and oxygen-18 concentrations during the simultaneous separation by vacuum distillation of water.

PARAMETERS OF SEPARATION OF HYDROGEN AND OXYGEN ISOTOPES IN THE NONSELECTION MODE

The separation of hydrogen and oxygen isotopes by distillation was experimentally studied during the testing of high-performance stainless steel packings made at the Mendeleev University of Chemical Technology of Russia. The experiments were performed on experimental stands, which included distillation columns with a diameter of 60, 120, and 250 mm and a height of packing of $H = 1$ m; evaporator and condenser—accordingly, the lower (LFRU) and upper (UFRU) flow reversal units; and systems for selection, evacuation, control, and automatic control over the process parameters [9, 10]. The unit with a column with a diameter of 250 mm was equipped with a heat recovery system using a heat pump based on the freon cycle [11]. The studies were performed using deionized water of natural isotopic abundance. The isotope analysis of the samples was performed using a T-LWIA-45-EP spectral liquid isotope analyzer designed for analysis of water at the level of the natural isotopic abundance with a measurement error of ±0.0001 at % (1 ppm).

The separation degree (K), the number of theoretical plates (NTP, N), and the height equivalent to a theoretical plate (HEPT, h) were calculated from the results of the analysis of deuterium or ^{18}O concentrations at the bottom (x_b) and top (x_t) of the column by the following equations [1, 3]:

$$K = \frac{x_b(1 - x_t)}{x_t(1 - x_b)}, \tag{8}$$

$$N = \frac{\ln K}{\ln \alpha}, \tag{9}$$

$$h = \frac{H}{N}. \tag{10}$$

Tables 2 and 3 present the mass transfer characteristics of the packings in the nonselection mode at a pressure of 0.02 MPa and specific load of the liquid (L_{sp}, kg/(m^2 h)) of 80% of the limiting load. The samples of the spiral-prismatic packing (SPP) with elements with dimensions of 4 × 4 × 0.25, 6 × 6 × 0.4, and 8 × 8 × 0.4 mm were tested (Table 2). Table 3 shows the results of tests of a regular rolled ribbon-screw packing (RRSP) with a height of 1000 mm made of corrugated stainless mesh of 0.76 mm from wire of 0.27 mm 12Kh18N10T with the following characteristics: straight type of corrugation of original ribbons; width of original tape 47 mm; width of corrugated strip 40 mm; height of grid corrugation for winding the block 5 mm; winding with a gap of 3 mm between the turns; layer of four parallel strips. “Skirts” of straight stainless mesh ribbons were mounted at a distance of 200 mm from one another along the height of the block; the block diameter was ~10 mm smaller than the shell diameter for the “skirts” in a straightened state to provide contact between the packing and the shell wall when mounted in the column. The packing was loaded, initiated, and processed by the original procedures developed at the Mendeleev University of Chemical Technology of Russia [9, 10].

According to the presented data, the increase in the throughput capacity and HEPT for SPP are approximately proportional to the increase in the size of the packing element. At the same time, a twofold increase in the column diameter leads to a 1.11-fold increase in HEPT on average, i.e., to an increase in proportion to the ratio of diameters to the power of 0.17. This agrees well with the literature data, according to which the exponent recommended for evaluating the scale transition factor for small highly effective packings with uniform multipoint refluxing was taken to be 0.2 [3]. For RRSP, the scale transition factor expressed as the ratio of HEPT is proportional to the ratio of column diameters to the power of 0.156.

The most significant result from the viewpoint of the study of concentration profiles during separation is that the HEPT values obtained for the simultaneously separated H_2O–HDO and $H_2^{16}O - H_2^{18}O$ isotopic systems coincide with high accuracy. This suggests that in the nonselection mode, the concentration profiles of deuterium and oxygen-18 will be the same.

Table 2. Mass exchange characteristics of SPP during vacuum water distillation at a pressure of 0.02 MPa

Isotope system	α	L_{sp}, kg/(m^2 h)	Heavy isotope concentration, ppm		K	h, cm
			x_b	x_t		
Column with a diameter of 60 mm						
$Z = 4$ mm						
H_2O–HDO	1.046	1600	142	45	3.16	3.9
$H_2^{16}O$–$H_2^{18}O$	1.006		1965	1692	1.16	4.0
$Z = 6$ mm						
H_2O–HDO	1.046	2450	141	74	1.90	7.0
$H_2^{16}O$–$H_2^{18}O$	1.006		1964	1801	1.09	6.9
Column with a diameter of 120 mm						
$Z = 4$ mm						
H_2O–HDO	1.046	1600	140	49	2.84	4.3
$H_2^{16}O$–$H_2^{18}O$	1.006		1966	1699	1.16	4.1
$Z = 6$ mm						
H_2O–HDO	1.046	2450	140	74	1.88	7.1
$H_2^{16}O$–$H_2^{18}O$	1.006		1965	1810	1.08	7.3
$Z = 8$ mm						
H_2O–HDO	1.046	3100	141	92	1.53	10.5
$H_2^{16}O$–$H_2^{18}O$	1.006		1965	1855	1.06	10.4

Table 3. Mass exchange characteristics of RRSP during vacuum water distillation at a pressure of 0.02 MPa

Isotope system	α	L_{sp}, kg/(m^2 h)	Heavy isotope concentration, ppm		K	h, cm
			x_b	x_t		
Column with a diameter of 60 mm						
H_2O–HDO	1.046	5280	140	106	1.320	16.2
$H_2^{16}O$–$H_2^{18}O$	1.006		1965	1893	1.038	16.0
Column with a diameter of 120 mm						
H_2O–HDO	1.046	5440	141	109	1.285	17.9
$H_2^{16}O$–$H_2^{18}O$	1.006		1965	1901	1.033	18.2
Column with a diameter of 250 mm						
H_2O–HDO	1.046	5420	142	114	1.250	20.2
$H_2^{16}O$–$H_2^{18}O$	1.006		1966	1907	1.031	19.8

DEVELOPMENT OF AN ALGORITHM FOR CALCULATING THE CONCENTRATION PROFILES OF HYDROGEN AND OXYGEN ISOTOPES IN THE PRODUCT SELECTION MODE

From a practical viewpoint, it is important to determine the distribution of isotope concentrations during the simultaneous separation of H_2O–HDO and $H_2^{16}O - H_2^{18}O$ mixtures in the product selection mode for the “closed” scheme (Fig. 1). The calculation algorithm was developed using the general theory of fine separation of mixtures for low concentrations of the heavy isotope [1].

At the first stage, the external flows and the concentrations of the target isotope (for example, deute-

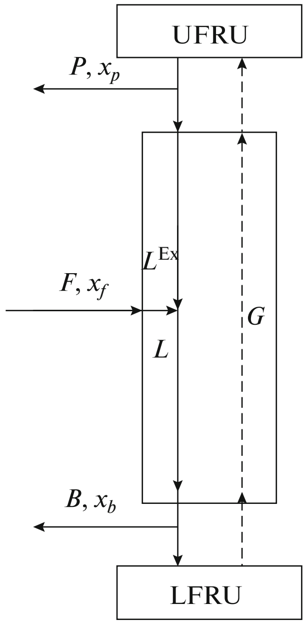

Fig. 1. Schematic diagram of the column operating according to the “closed” scheme.

rium) in them are determined from the initial data using the material balance equations:

$$F = P + B, \tag{11}$$

$$Fx_f = Px_p + Bx_b, \tag{12}$$

where x_f, x_p, and x_b are the concentrations of the heavy isotope (au) in the feed (F), waste (P), and product (B) flows, respectively.

The working pressure (temperature) at the top and bottom of the column was set with allowance for the hypothetical hydraulic resistance; for the average value, the separation factor was calculated for the H–D isotope system by Eqs. (4) and (6). Then the relative selection (θ) was given arbitrarily.

The concentrating section of the separation unit is calculated by the following recurrent equations:

$$K_{\mathrm{C}} = \frac{x_b(1-x_f)}{x_f(1-x_b)}, \tag{13}$$

$$\lambda_{\mathrm{C\,min}} = \frac{x_b - x_f}{x_b - \dfrac{x_f}{\alpha - \varepsilon x_f}}, \tag{14}$$

$$L_{\mathrm{C\,min}} = \frac{B\left(x_b - \dfrac{x_f}{\alpha}\right)}{\dfrac{\varepsilon}{\alpha} x_f(1-x_f)}, \tag{15}$$

$$\theta = \frac{L_{\mathrm{C\,min}}}{L_{\mathrm{C}}} = \frac{1-\lambda_{\mathrm{C}}}{1-\lambda_{\mathrm{C\,min}}}, \tag{16}$$

$$G = L - B, \tag{17}$$

$$N_{\mathrm{C}} = \frac{\ln \dfrac{K_{\mathrm{C}}(\alpha-1)\lambda_{\mathrm{C}}}{\alpha - \lambda_{\mathrm{C}} - K_{\mathrm{C}}(1-\lambda_{\mathrm{C}})\alpha}}{\ln \dfrac{\alpha}{\lambda_{\mathrm{C}}}}, \tag{18}$$

where K_{C} is the degree of separation; $\lambda_{\mathrm{C\,min}}$, λ_{C} are the minimum and real molar ratios of the flows; $L_{\mathrm{C\,min}}$, L_{C} are the minimum and real molar flows of the liquid; G is the molar vapor flow; and N_{C} is the number of the theoretical plates.

Then we calculate the exhaustive section of the column:

$$K_{\mathrm{Ex}} = \frac{x_f(1-x_p)}{x_p(1-x_f)}, \tag{19}$$

$$L_{\mathrm{Ex}} = L - F, \tag{20}$$

$$\lambda_{\mathrm{Ex}} = \frac{G}{L_{\mathrm{Ex}}}, \tag{21}$$

$$\lambda_{\mathrm{Ex\,max}} = \frac{x_f - x_p}{\dfrac{x_f}{\alpha - \varepsilon x_f} - x_p}, \tag{22}$$

$$N_{\mathrm{Ex}} = \frac{\ln[K_{\mathrm{Ex}}(1-\theta)]}{\ln \dfrac{\alpha}{\lambda_{\mathrm{Ex}}}}, \tag{23}$$

where K_{Ex} is the degree of separation; $\lambda_{\mathrm{Ex\,max}}$, λ_{Ex} are the maximum and real molar ratios of the flows; L_{Ex} is the molar liquid flow; and N_{Ex} is the number of theoretical plates.

The concentration of the target isotope in the vapor flow (y) in any section of the column is calculated using the equations of working lines for the concentrating or exhaustive sections of the column:

$$x = \lambda_{\mathrm{C}} y + (1-\lambda_{\mathrm{C}})x_b, \tag{24}$$

$$x = \lambda_{\mathrm{Ex}} y + (\lambda_{\mathrm{Ex}} - 1)x_p. \tag{25}$$

Then optimization with respect to θ can be performed using the minimum value of the total flow (J) as the optimization parameter that defines the volume of the separating equipment:

$$J = (L_{\mathrm{C}}N_{\mathrm{C}} + L_{\mathrm{Ex}}N_{\mathrm{Ex}}). \tag{26}$$

To calculate the concentration profile of the second isotope system (for example, ^{16}O–^{18}O), as the initial parameters we use the oxygen concentration in the feed flow $x_{f,\mathrm{O}}$, the separation factor calculated by Eqs. (5) and (7) based on the process conditions, and the flow values obtained by calculating the target system.

Table 4. Parameters of the simultaneous separation of hydrogen and oxygen isotopes during water distillation (pressure 0.03 MPa, $F = 1$ kmol/h, $B = 0.5455$ kmol/h, $P = 0.4545$ kmol/h)

Parameter	Concentrating section		Exhausting section	
	H_2O–HDO	$H_2^{16}O - H_2^{18}O$	H_2O–HDO	$H_2^{16}O - H_2^{18}O$
Relative selection θ	0.78	0.5836735	0.78	0.5836735
Molar ratio of flows λ	0.896868		1.10576	
Water flow L, kmol/h	5.2977		4.2977	
Degree of separation	1.25	1.0263	1.4286	1.033
Concentration at the top of the column, at. fractions	0.01	0.001966	0.007	0.00190387
Concentration at the bottom of the column, at. fractions	0.0125	0.002018	0.01	0.001966
Number of theoretical plates N	11.7	7.8	18.8	8.9

Given an arbitrary value of the relative selection (θ_O), the degree of separation in the concentrating part of the unit (K_{CO}) is determined by the equation

$$1 - \theta_O = \frac{K_{CO} - 1}{K_{mCO} - 1} \qquad (27)$$

using the previously obtained value of N_K and the maximum degree of separation (K_{mCO}) calculated by Eq. (9).

Then the column separation parameters are calculated for the system using Eqs. (13)–(16). The calculation is performed by iterations, while varying the θ_O values, until the value of the molar ratio of flows ($\lambda_{C,O}$) for the system coincides with the value (λ_C) previously calculated for the target isotope. After that, the NTP ($N_{C,O}$) required to achieve the heavy isotope concentration equal to $x_{b,O}$ in the B flow is calculated by Eq. (18). The heavy isotope concentrations in the vapor flow ($y_{f,O}$) in the section of the feed inlet point and in the waste ($x_{p,O}$) are determined by the equations of the working lines (24) and (25), respectively. Then the degree of separation and NTP in the exhausting part of the column are determined by Eqs. (19) and (23), respectively.

Let us consider the problem of determining the concentration profile of deuterium and oxygen-18 in a column that operates according to the "closed" scheme under the following conditions: deuterium concentration $x_f = 0.01$ in the feed flow, $x_b = 0.0125$ in the concentrate, and $x_p = 0.007$ in the waste; feed flow $F = 1$ kmol/h; oxygen-18 concentration in the flow F $x_{f,O} = 0.001966$; and the average pressure in the column 0.03 MPa ($T_{av} = 342$ K), for which the separation factors were $\alpha_{H-D} = 1.040$ and $\alpha_{16O-18O} = 1.00568$. The optimum relative selection for the H–D system was $\theta = 0.78$. The parameters of the column are presented in Table 4. If we assume that the column is filled with RRSP, whose characteristics are presented in Table 3 and [9], the column diameter is 150 mm, and the height of the concentrating and exhaustive sections (at $h = 18$ cm) is 2.1 and 3.4 m, respectively.

According to the presented data, the separation parameters of these isotope mixtures differ significantly, and the NTP required to achieve balance concentrations in the concentrate and waste for ^{16}O–^{18}O is smaller than for the H–D system. This is confirmed by the "plate-by-plate" calculation, the results of which in the form of NTP are presented in the y–x diagram in Fig. 2.

The expected profiles of deuterium and oxygen-18 concentrations obtained by this method in the concentrating and exhausting parts of the column are presented in Fig. 3.

According to Fig. 3, some plates in both the exhausting and concentrating sections are not involved in the separation. Note that a similar dependence was observed during the detritiation of off-grade heavy water by chemical isotope exchange in the water–hydrogen system at a pilot unit at the Mendeleev University of Chemical Technology of Russia: despite the fact that the ratio of the heights of the concentrating and exhaustive parts was 1 : 5, the whole unit was filled with concentrated heavy water, and the deuterium concentration decreased only in the last exhausting column. The profile of tritium concentration corresponded to the calculated parameters in this case [12]. Thus, it can be expected that in our case, the nontarget ^{18}O isotope will also be redistributed toward

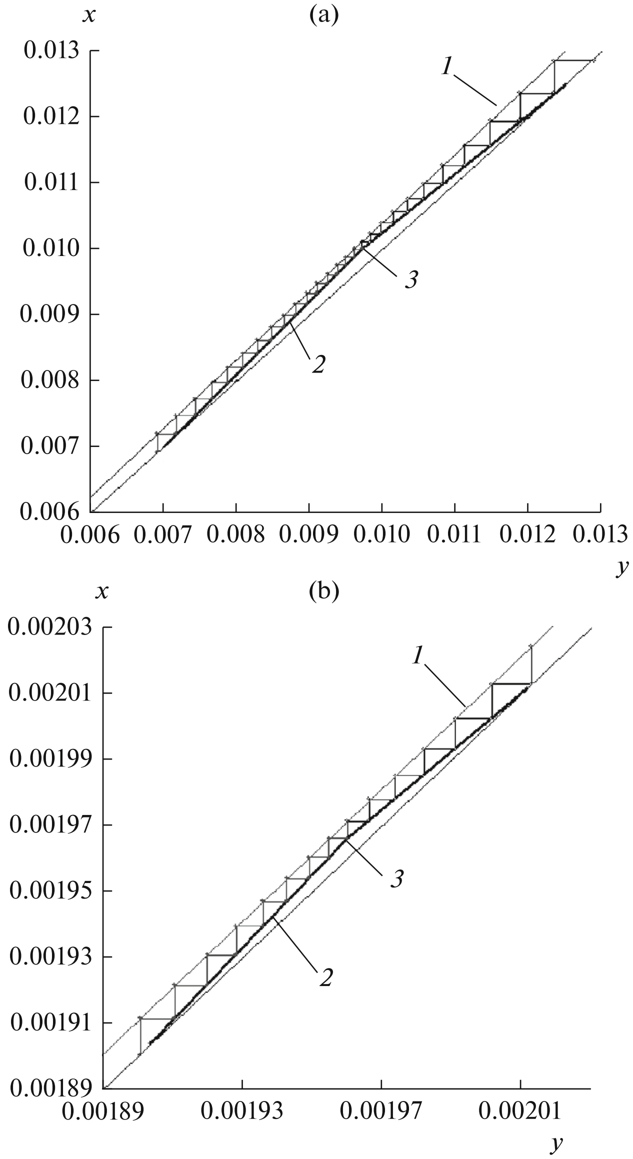

Fig. 2. Working diagram y–x of a unit for separation of (a) hydrogen and (b) oxygen isotopes: (*1*) equilibrium line, (*2*) working line, and (*3*) feed inlet point.

an increase in the concentrating part, and the concentrations of oxygen isotopes will be mixed at the feed inlet point. Figure 4 shows the predicted concentration profile of ^{18}O along the height of the column constructed in an assumption that the origin of NTP is at the upper end of the column and the concentration mixing is neglected.

According to Fig. 4, the predicted concentration profile of ^{18}O differs significantly from the calculated one and from the profile of deuterium. At the feed inlet point, the difference in concentrations of ^{18}O in the internal liquid flow and the F flow will differ in proportion to the degree of separation K_C. Thus, it can be assumed that the exhausting part of oxygen-18 will decrease with the increasing degree of deuterium concentration.

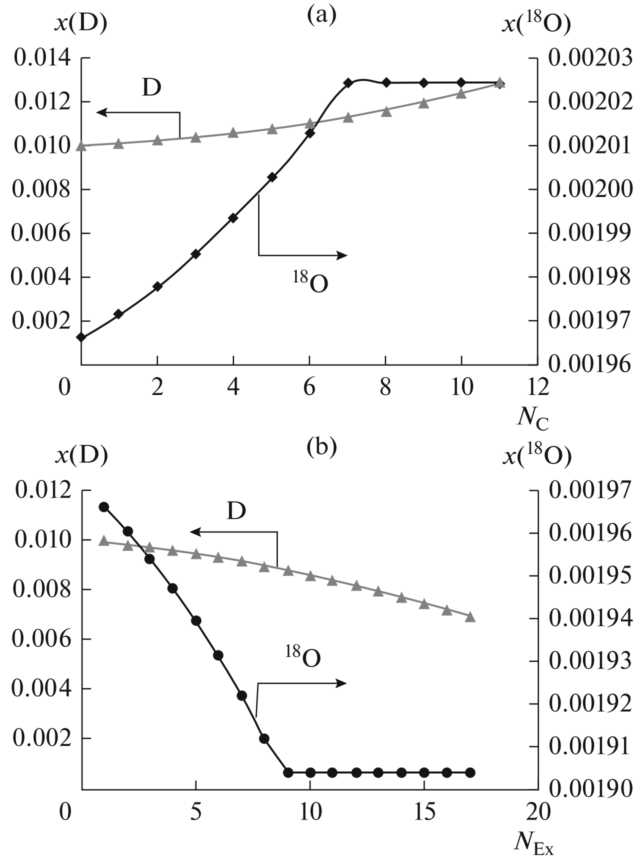

Fig. 3. Concentration profiles of deuterium and oxygen-18 in the (a) concentrating and (b) exhausting parts of the column.

CONCLUSIONS

Based on the above data, it can be concluded that the number of theoretical plates in a column with upper and lower flow circulation units can be determined with simultaneous separation of hydrogen and oxygen isotopes in the nonselective mode.

An algorithm was developed for evaluating the concentration profile of hydrogen and oxygen isotopes during their simultaneous separation in a distillation column based on the principles of the general theory of fine separation of mixtures for the range of low concentrations. It was shown, using the simultaneous separation of isotopes in H_2O–HDO and $H_2^{16}O$–$H_2^{18}O$ mixtures as an example, that in a column operating according to the “closed” scheme with product selection, the relative selection and the number of theoretical plates for deuterium and oxygen-18 are different.

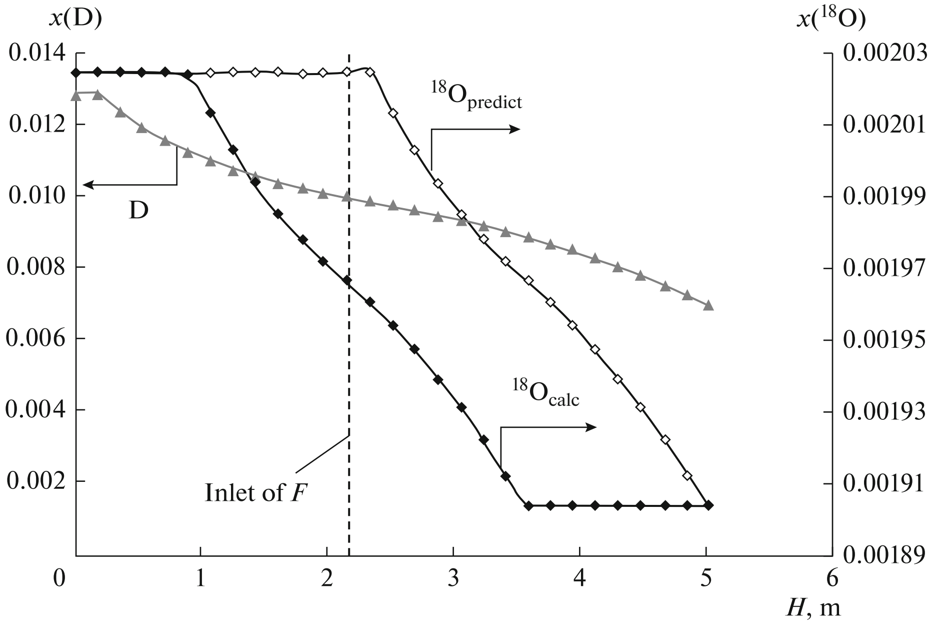

Fig. 4. Concentration profiles of deuterium and oxygen-18 along the height of the column.

A consequence of this are the different dependences of the concentration profile of these isotopes along the height of the column.

ACKNOWLEDGMENTS

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NOTATION

G	molar vapor flow, kmol/h
H	height of the packed part of the column, m
h	height equivalent to a theoretical plate, cm
J	minimum value of the total flow, kmol/h
K	degree of separation
L	liquid flow, kmol/h
L_{sp}	specific load of liquid, kmol/h
N	number of theoretical plates
x	heavy isotope (D or ^{18}O) concentration in liquid water, at. fractions
y	heavy isotope (D or ^{18}O) concentration in the vapor, at. fractions
α	separation factor
ε	enrichment coefficient
θ	relative selection
λ	molar ratio of flows

SUBSCRIPTS AND SUPERSCRIPTS

0	initial value
b	product flow
f	feed flow
max (m)	maximum
p	waste flow
t	top
Ex	exhausting section of the column
C	concentrating section of the column
b	bottom
sp	specific

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