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Efficiency of Mixture Separation in Distillation Columns with Structured Packings under Conditions of Dynamically Controlled Irrigation

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The paper presents the results of an experimental study of separation efficiency and distribution of flow parameters over the cross-section of the distillation columns. The experiments were carried out on the large-scale research setup “Large Freon Column” with the diameter of 0.6 and 0.9 m. Experimental data were obtained at uniform and non-uniform irrigation of the Mellapak 350.Y and Sulzer 500X structured packings by the controlled liquid distributor. The mixture of refrigerants R114 and R21 was used as the working liquid at the pressure of 3 bar in the column. The liquid distributor with independently controlled valves for each drip point was used for controlled packing irrigation. Local temperature distributions of the liquid phase and counter-current vapor flow in the cross-section and over the packing height were recorded using a sequence of fast-response temperature sensors. Periodic irrigation of the structured packing can affect large-scale non-uniformity of mixture composition and flow parameter distribution over the cross-section and height of the transfer unit. Significantly non-uniform packing irrigation within a separate semiperiod leads to rearrangement of the structure of large-scale non-uniformity inside the packing.
The new method for dynamically controlled irrigation of the packing is suggested. This method is aimed at destruction of stationary large-scale maldistribution over the column cross-section and achievement of higher separation efficiency of a completely wetted packing.

1. Introduction

Distillation columns are widely used in petroleum, chemical and food industries to separate the mixtures of various liquids. The basic requirement for the effective operation of distillation column is uniform distribution of the liquid film over the packing and the counter-current vapor flow over the column cross-section. To achieve this, the packing of metal sheets with microtexture of different scales and highly permeable porous packing were designed (Pavlenko et al., 2016, A). In recent years, in order to increase the production capacity and improve separation efficiency at distillation, the scientists have performed a lot of study on the structured packing (Pavlenko et al., 2010; Soulaine et al., 2014; Olujic et al., 2015). These researches were mainly focused on the improvement of packing structure, special treatment of packing surface and organization of the optimal structure of irrigation points in the liquid distributor. Authors (Pavlenko et al., 2017, A) carried out experimental study on the influence of microtexture, holes, inclination angle of the large ribs on the parameters of the liquid spreading on single elements of structured packings at different irrigation density. The use of the modern structured packings allows more uniform distribution of a liquid film over the packing surface, but it does not completely solve the problem of uniform distribution of the flows along the entire height of the packings in real conditions of mixtures separation in distillation columns.
Stationary irrigation of the structured packing in large-scale industrial distillation columns is usually performed by the liquid distributors with a fixed drip point density over the column cross-section. For stationary setting of irrigation points under the conditions of the liquid film flow over the structured packing and the counterflow vapor flow, very significant large-scale maldistributions of the flow density develop over the packing cross-section and height. Maldistribution significantly reduces the efficiency of mixture separation in distillation

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columns of the large diameter. Pavlenko et al. (2014) noted that a negative vapor stratification along the packing height caused by different densities of vapor mixture components and higher temperature at the column bottom can lead to formation of large-scale maldistribution of temperature and mixture compositions over the column cross-section even at uniform packing irrigation by liquid. The idea of unsteady packing irrigation using controlled liquid distributor for destruction of such stationary large-scale non-uniformities of local liquid and vapor flow rates, and mixture composition over the column cross-section was proposed by the authors of this work. Egger and Fieg (2017) have showed that short-term perturbations in the feed flow rate and thermal power allow maintaining the stable and safe operation of reactive DWC, which is important for successful adaptation of such columns in the chemical industry. The distillation column is an operation unit with multiple controlled and manipulated variables. There are some controlled variables in the distillation column: distillate and bottom product composition, column pressure, liquid level inside an accumulator and liquid level in the bottom column. There are also some manipulated variables: distillate flow rate, bottom product flow rate, reflux flow rate and heat duty for condenser and reboiler. The PID controller cannot solve the industrial process complexity and the quality of product specification. Haura et al. (2017) have developed the Internal Model Control for simulation of both steady state and dynamic vacuum distillation.

The pulsating supply of the liquid flow (Memedlyaev et al., 1994) makes it possible to create phase distribution in the packed column, when the height of transfer unit becomes significantly less than that under the stationary conditions of irrigation.

In the experiments presented below, the method of destruction of stationary large-scale non-uniformities of local liquid and vapor flow rates, and mixture composition over the column cross-section was implemented using a dynamically controlled liquid distributor.

2. Setup and method description

The experiments were carried out at the research setup Large Freon Column, designed to study the efficiency of mixture separation and distribution of local flow parameters during the distillation process on the structured packings of various types. This setup allows working with the packing up to 0.9 m in diameter and up to 4 m in height. The working mixture R114/R21 was chosen from the condition of modeling the process of cryogenic mixture rectification. The efficiency of mixture separation by the packing was determined by chromatographic analysis of mixture composition in the liquid and vapor phases at the column inlet and outlet. The detailed description of the applied methods and design of the research setup is given by Pavlenko et al. (2014).

The experiments were carried out on two types of the structured packing – Mellapack 350.Y and Sulzer 500X. The Mellapack 350.Y packing had a diameter of 0.9 m, and the Sulzer 500X packing had a diameter of 0.6 m. The packing consisted of 10 layers and its height was 2.1 m and 2.2 m, respectively. To irrigate the packing, a liquid distributor specially developed by the authors was used. The height of the vessel of liquid distributor was 500 mm, and this allowed us to work at different flow rates within a 3-fold range. In the bottom of the distributor, there are the holes, where the nozzles of the desired diameter are installed (3 - 5 mm). The holes are arranged in parallel rows at the distance of 99 mm. Over the rows of holes, there are the mounting rails, which allow the fixing of solenoid valves above the holes for nozzle blocking. In the case of the Mellapack 350.Y packing with the diameter of 0.9 m, the valves in the row were installed at the distance of 44 mm; for the Sulzer 500X packing with the diameter of 0.6 m, they were installed at the distance of 22 mm. Each valve was controlled independently both manually and automatically (Pavlenko et al., 2017, B). The layout of controlled drip points of the liquid distributor for the packing with the diameter of 0.9 and 0.6 m is shown in Figure 1.

To control the uniformity of temperature distribution inside the packing, the semiconductor thermometers were installed in three packing cross-sections at different heights. In each cross-section, there were 16 thermometers. The thermometers were mounted on the upper edge of the 2nd, 5th and 8th packing layers, counting from the bottom of the column. All thermometers was sampled during 4-5 s. Data on maldistribution degree and nature of temperature distribution were displayed in real time on the display in the form of topograms of temperature distribution for each cross-section and value of standard deviation for 16 temperature measurements in each cross-section.

The experiments were carried out at steady-state irrigation with uniform drip point distribution over the column cross-section and periodically changing structure of the irrigation points, conventionally called the *HC* pattern. The *HC* pattern for the Sulzer 500X packing with 0.6-m diameter provides irrigation of a half of packing cross-section during time t_1, the second half of cross-section stays closed. Then, the valves are switched, and the irrigated and dry halves of cross-section change for period t_2. Then, the cycle is repeated periodically. The main data array for the *HC* pattern is obtained for equal values of t_1 and t_2 within 5 - 160 s. The pictures of packing irrigation by the *HC* pattern are shown in Figure *1*. The Mellapack 350.Y packing with the diameter of 0.9 m was periodically irrigated in one and another half of the column cross-section in the same manner. The layout of liquid jets at uniform and periodic irrigation of the 350.Y packing is given by Pavlenko et al. (2016, B).

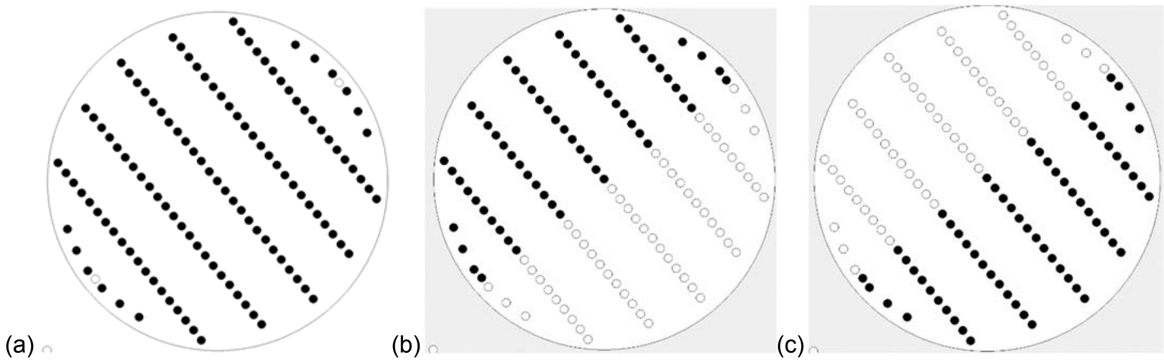


Figure 1: Drip point location: a – uniform irrigation; b – first semiperiod of periodic irrigation, t_1, HC pattern; c – second semiperiod of periodic irrigation, t_2, HC pattern. Black point's – open, white point's – close.

3. Results and discussion

The experiments with periodic irrigation of the packing were carried out under the conditions of complete reflux in the range of superficial vapor velocity 0.612 < *F-factor* < 1.300 $Pa^{0.5}$ (*F-factor* = $U_v(\rho_v)^{0.5}$). Dependence of the height of transfer unit (*HTU*) on the value of packing irrigation period is shown in Figure *2* for different superficial vapor velocities. Efficiency of mixture separation in the column [height of transfer unit (HTU) and HETP] is calculated using the the measured mixture composition in the vapor and liquid phases at the column inlet and outlet and equilibrium data. The calculation procedure is given by Pavlenko et al. (2014).

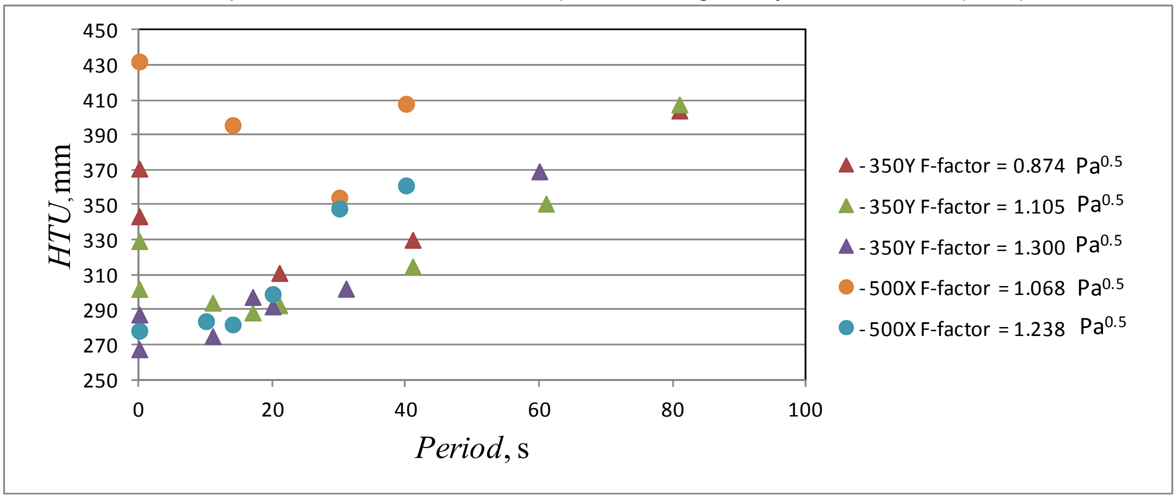


Figure 2: HTU vs. irrigation period t = t_1+ t_2. Uniform irrigation – t = 0.

As it can be seen from the diagram, at periodic packing irrigation according to the *HC* pattern in the range of *F-factor* < 1.105 $Pa^{0.5}$, an increase in the efficiency of mixture separation was observed with an increase in the switching period; then, separation efficiency became worse for both types of the packing. At switching with a short period, separation efficiency was close to separation efficiency at uniform irrigation because during irrigation of one half of the packing cross-section, there was not time for liquid to penetrate into a significant part of the packing height. In fact, switching irrigation to another half of the packing cross-section created the conditions of more uniform irrigation. An increase in the switching period led to the fact that during one semiperiod of irrigation liquid could spread over the packing height, and the large-scale non-uniformity corresponding to the conditions of non-uniform irrigation during the first semiperiod, started forming in the packing. At that time, switching to the second irrigation semiperiod, which is diametrically opposite by the non-uniformity structure to the first semiperiod, led to rearrangement of the formed large-scale non-uniformity in the opposite direction. As a result, for switching period t_{min}, the maximal efficiency of mixture separation was achieved. It exceeded separation efficiency at uniform irrigation by 20%. The value of optimal period t_{min} is determined by the time of liquid penetration along the packing height, and it depends on both the packing height and superficial velocity of liquid and vapor flows.

The diagram of a change in the standard deviation of temperature over the 2nd layer of the Sulzer 500X packing for superficial vapor velocity *F-factor* = 1.238 $Pa^{0.5}$ (*HC* periodic irrigation) is shown in Figure *3*. It can be seen from the diagram that minimal non-uniformity and minimal change in temperature maldistribution are observed in the regime with a period of 14 s. An increase in the period leads to an increase in the degree of temperature maldistribution in the packing layer. It is seen in Figure 2 that for a period longer than 14 s, the efficiency of mixture separation deteriorates.

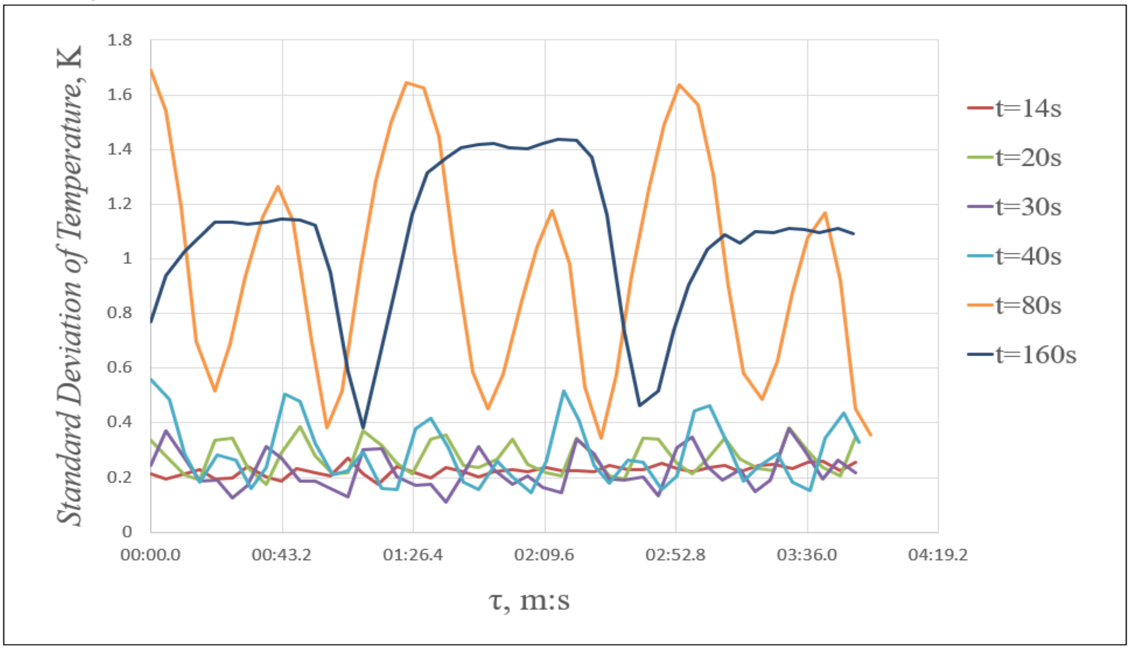


Figure 3: Time dependence of the value of temperature standard deviation in the 2nd layer of the Sulzer 500X packing at different duration periods t.

The character of a change in temperature distribution in the cross-section of the Sulzer 500X packing at static uniform and periodic irrigation *HC* with the period of 30 s and *F-factor* = 1.238 $Pa^{0.5}$ is shown in Figure 4 and Figure 5*.*

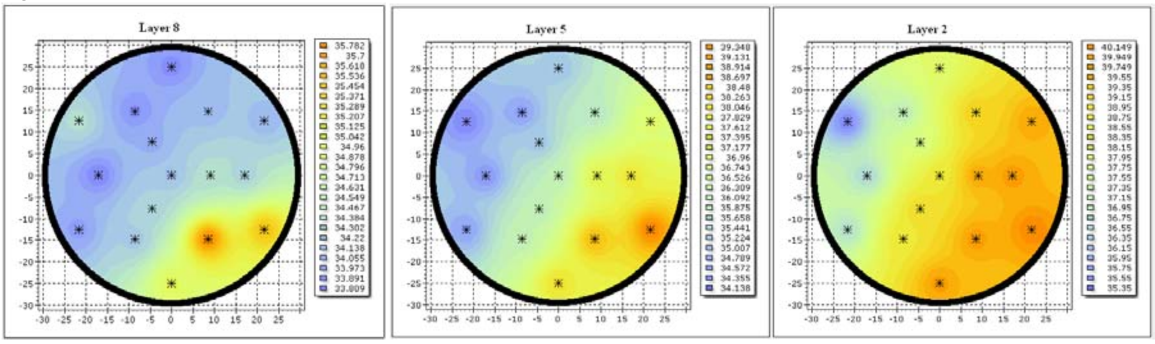
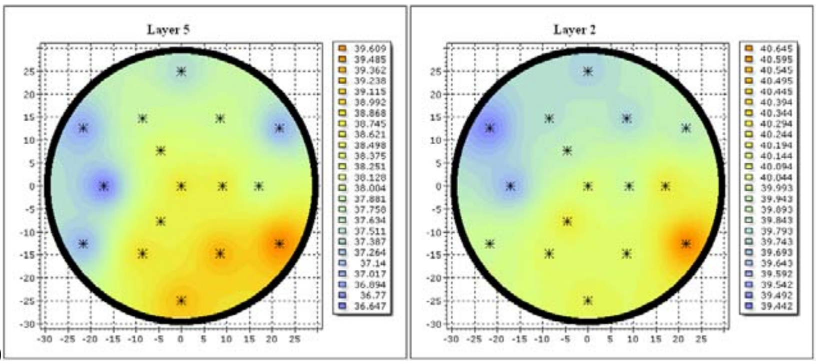


Figure 4: Temperature distribution in the packing cross-section at static uniform irrigation.

At static irrigation with a uniform drip point pattern (Figure 1a), large-scale temperature maldistribution is formed over the column cross-section in the packing, which extends along the entire column height (Figure 4). At that, the position of the regions with maximal and minimal temperature in the cross-section is kept throughout the entire height of the column. The asterisks show the location of thermometers in the packing cross-section. At dynamic irrigation, irrigation is highly uneven during each individual semiperiod. Therefore, when switching from one semiperiod to another, distribution of the local flow parameters inside the packing changes. A change in the character of temperature distribution across the packing cross-section at the level of the 5th and 2nd layers during one period of the cycle of switching the liquid distributor valves is shown in Figure 5. The figures show standard deviations StDev_5 and StDev_2 of 16 thermometers in the 5th and 2nd layers, respectively.



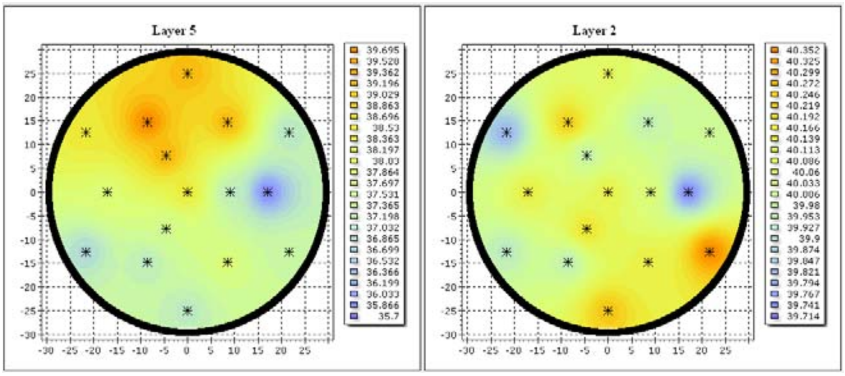
(a)

τ_1 = 0 s;

StDev_5 = 0.92 K;

StDev_2 = 0.30 K.

(b)

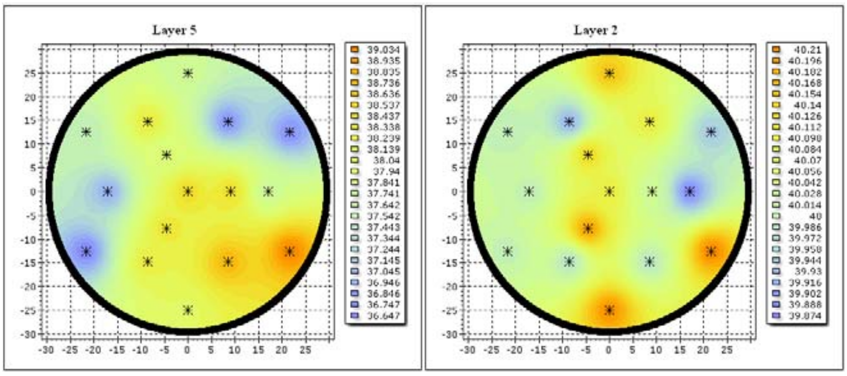


τ_1 = 11 s;

StDev_5 = 1.14 K;

StDev_2 = 0.17 K.

(c)

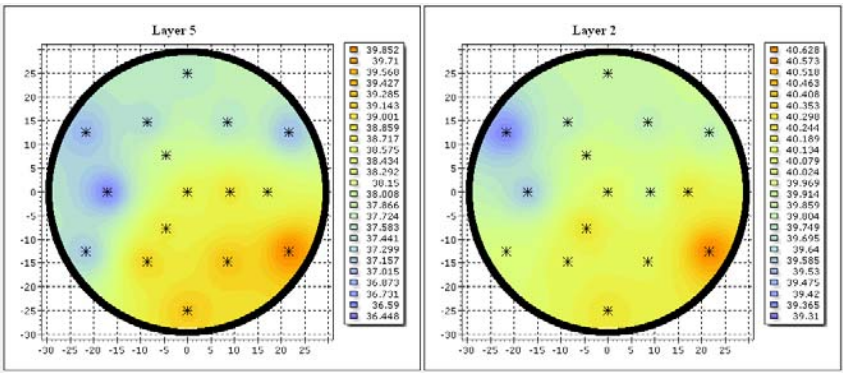


τ_1 = 21 s;

StDev_5 = 0.75 K;

StDev_2 = 0.11 K.

(d)



τ_1 = 32 s;

StDev_5 = 1.04 K;

StDev_2 = 0.31 K.

Figure 5: The character of temperature distribution in the packing cross-section at periodic irrigation with the period of 30 s. τ_1 – time from period beginning.

As it can be seen from the above-mentioned topograms, the character of large-scale temperature maldistribution changes during the valve-switching period. At that, rearrangement is asymmetric, despite switching of liquid distributor valves, symmetric in time and space. The least degree of temperature maldistribution in the packing cross-section in dynamic irrigation regime is observed. Apparently, under these conditions, the most uniform distributions of local liquid and vapor flow rates over the cross-section and height of the structured packing are achieved.

The additional series of experiments showed that minimal correction of the drip point pattern of liquid distribution could significantly affect distribution of the flow parameters over the cross-section and height of the mass transfer unit and increase separation efficiency of the column within 20%. When column operation became quasistationary and the structure and degree of large-scale temperature maldistribution over the column cross-section were registered, a minimal correction of the drip point pattern in liquid distribution was made. This correction was made by opening one hole in the liquid distributor in the region of projection of the large-scale zone of maximal temperatures with simultaneous closing of one hole in the region of projection of the minimal temperature zone. As a result, the structure and degree of large-scale temperature maldistribution over the column cross-section and, consequently, the efficiency of mixture separation were changed. By means of two or three above iterations, a minimal degree of temperature maldistribution in the column cross-sections was achieved and, as a consequence, the best efficiency of mixture separation.

4. Conclusions

According to the experiments, the influence of periodic irrigation on the large-scale non-uniformity of mixture composition, formed in the packing, can significantly change the structure of flow parameter distribution over the cross-section and height of the mass transfer unit. Essentially non-uniform periodic irrigation of the packing can improve separation efficiency of the column within 20%, if the switching periods are comparable with the times of formation of large-scale non-uniformity.

The experiments showed that minimal correction of the drip point pattern of liquid distribution can significantly affect distribution of the flow parameters over the cross-section and height of the mass transfer unit and increase the separation efficiency of the column within 20%.

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