TRANSFER BY METALLIC SERVICE LINES AND SORPTION EXTRACTION OF MoF$_6$ FROM THE GAS FLOW

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The results of investigation of the sorption of molybdenum hexafluoride MoF$_6$ on granulated sodium fluoride NaF have been presented; the data of measurements of the MoF$_6$ loss in transport by technological service lines of a reactor ampoule plant with molten-salt fuel have been given.

Keywords: sorbent, radioisotopes, molten salts, molybdenum hexafluoride, Mo-99, sodium fluoride.

Introduction. A new method of producing radioisotopes (^{99}Mo, ^{89}Sr, and ^{133}Xe) for medical purposes in a liquid (molten)-salt nuclear fuel has been proposed in [1–3]. The aggregate state of the fuel based on Li, Be, and U fluorides offers the possibility of extracting ^{99}Mo, ^{89}Sr, and ^{133}Xe from the molten salt selectively, leaving aside both ^{235}U and the basic group of fission products. As a result radiochemical conversion of the irradiated uranium, which is characteristic of solid-fuel systems, is excluded; this must finally lead to a decrease in the yield of high-level radioactive waste and to a reduction in the flow rate of fissionable materials in the technological process of production of radioisotopes.

The proposed technology is based on the distinctive feature of the behavior of the so-called noble metals Mo, Tc, Nb, and Ru [4, 5]. As the molten salt reactor experiments (MSRE, Oak Ridge, USA) have shown, up to 50% of ^{99}Mo can escape from the molten-salt fuel and enter, as aerosols or fluorides, the gas covering the salt's free surfaces.

A possible mechanism of synthesizing volatile ^{99}Mo fluorides in a molten-salt fuel has been considered in [6, 7]. It was assumed that ^{99}MoF$_n$ molecules are formed in the melt by radiation-chemical processes; in bubbling of the fuel with inert gas, these molecules are captured by the gas bubbles and are removed to a free volume above the melt surface where they, interacting with radiolytic fluorine, are oxidized to the higher fluoride ^{99}MoF$_6$.

To check the physical principles of the new technology of ^{99}Mo production on an IR-8 research and development reactor we create a RAMUS experimental setup (reactor ampoule plant) with a molten-salt fluoride fuel [6]. It is planned to determine the efficiency of ^{99}Mo yield from the fuel, the possibility of delivering molybdenum fluorides from the reactor core to the concentration zone, and the maximum degree of ^{99}Mo extraction from the carrier-gas flow.

The present work seeks to determine the MoF$_6$ loss in transport by the technological service lines of the ampoule plant as a function of temperature and to study the sorption of MoF$_6$ from the flows of a gaseous mixture Ar + MoF$_6$) with a content of molybdenum hexafluoride from $5 \cdot 10^{16}$ to $2 \cdot 10^{12}$ cm^{-3} on granulated sodium fluoride.

RAMUS Reactor Plant. It is planned to place the plant in the beryllium reflector of the IR-8 reactor, where the neutron flux can reach $\approx 10^{13}$ cm$^{-2} \cdot$sec^{-1}.

Characteristics of the RAMUS Plant

Molar composition of the fuel compound, %	66LiF–33.9BeF$_2$–0.1UF$_4$
Enrichment with a ^{235}U isotope, %	90
Mass of uranium, g	10.1
Volume of the salt, cm^3	450
IR-8 estimated power, MW	8
Total energy release in the fuel salt at a reactor power of 8 mW, kW	3.2

Range of operating temperatures of the salt at a reactor power of 8 MW, $^{\circ}C$	613–680
Carrier gas	helium + Kr and Xe
Flow rate of the carrier gas, cm^3/sec	1.0
Pressure of the carrier gas above the salt surface, MPa	0.08
Structural materials	NP2 nickel, copper, and 12Kh18N10T steel
Material of the ^{99}Mo sorbent	sodium fluoride NaF

Forced evacuation of ^{99}Mo, ^{89}Sr, and ^{133}Xe radioisotopes from the molten-salt fuel will be effected by bubbling the melt with helium. On traversal of the service lines and filters, in which short-lived fission products decay and are precipitated, by the carrier gas, the target radioisotopes are trapped in precipitators, from which they are subsequently extracted.

Selection of the Procedure of Measurement of the MoF_6 Concentration in the Gas Flows. The main difficulty in measuring the concentration of molybdenum hexafluoride MoF_6 is its low content in the gaseous mixture (10^{13}–10^{14} cm^{-3}); this content corresponds to the actual concentration of ^{99}Mo in the RAMUS plant. To measure the MoF_6 concentration in the gaseous mixture we use an FSM-1202 Fourier spectrometer for medium and near infrared regions.

Using the Bouguer–Lambert–Beer law we compute the cross section of radiation absorption by an MoF_6 molecule ($\nu = 739.2$ cm^{-1}), which is $9.6 \cdot 10^{-18}$ cm^2. The pressure of the multipass cell with an optical-path length of 4.8 m in the FSM-1202 spectrometer makes it possible to measure MoF_6 concentrations in the gaseous mixtures of up to $2 \cdot 10^{12}$ cm^{-3}.

Sorbent. We select sodium fluoride for MoF_6 sorption in the RAMUS plant. The character of interaction between molybdenum hexafluoride and NaF has been investigated in [8, 9] but only in the region of fairly high concentrations where MoF_6 forms compounds of composition Na_2MoF_8 and $NaMoF_7$. The MoF_6-vapor pressure above the resulting complex salts is as follows:

0.044 and 0.38 mm Hg at 25 and $100^{\circ}C$ respectively above Na_2MoF_8;

3 and 51 mm Hg at the same temperatures above $NaMoF_7$.

For MoF_6 concentrations of (10^{13}–10^{14}) cm^{-3} in the gaseous mixture and a service-line temperature of 50–$60^{\circ}C$, we can expect the absorption of molybdenum hexafluoride on NaF by the chemisorption mechanism; i.e., the formation of thermally stable surface chemical compounds. The absorption maximum must shift to the region of lower temperatures, as the content of the volatile fluoride in the gases decreases.

In the experiments, we use, as the MoF_6 sorbent, granulated sodium fluoride produced at the All-Russia Scientific-Research Institute of Chemical Engineering by the method of colloidal-chemical synthesis. This method makes it possible to produce fluorine-containing sorbents with a high porosity, a large specific surface, and a great diversity of the pores [10].

Sodium hydroxide and carbonate are the starting products in synthesizing. Shaping of granules from the gel-like mass is effected by the matrix method with a gel humidity of 28–30%, which ensures the production of a high-grade sorbent.

The properties of fluorine-containing sorbents are largely influenced by the calcination temperature. As the temperature increases, we observe partial sintering of the sorbents, which is accompanied by the increase in their crush strength and the decrease in the relative porosity. Sodium fluoride used in the present work has been produced at a calcination temperature of $350^{\circ}C$. Fluoride gels are precipitated at room temperature and are dried at a temperature of 60–$80^{\circ}C$.

Structural-Mechanical Characteristics of Sodium Fluoride

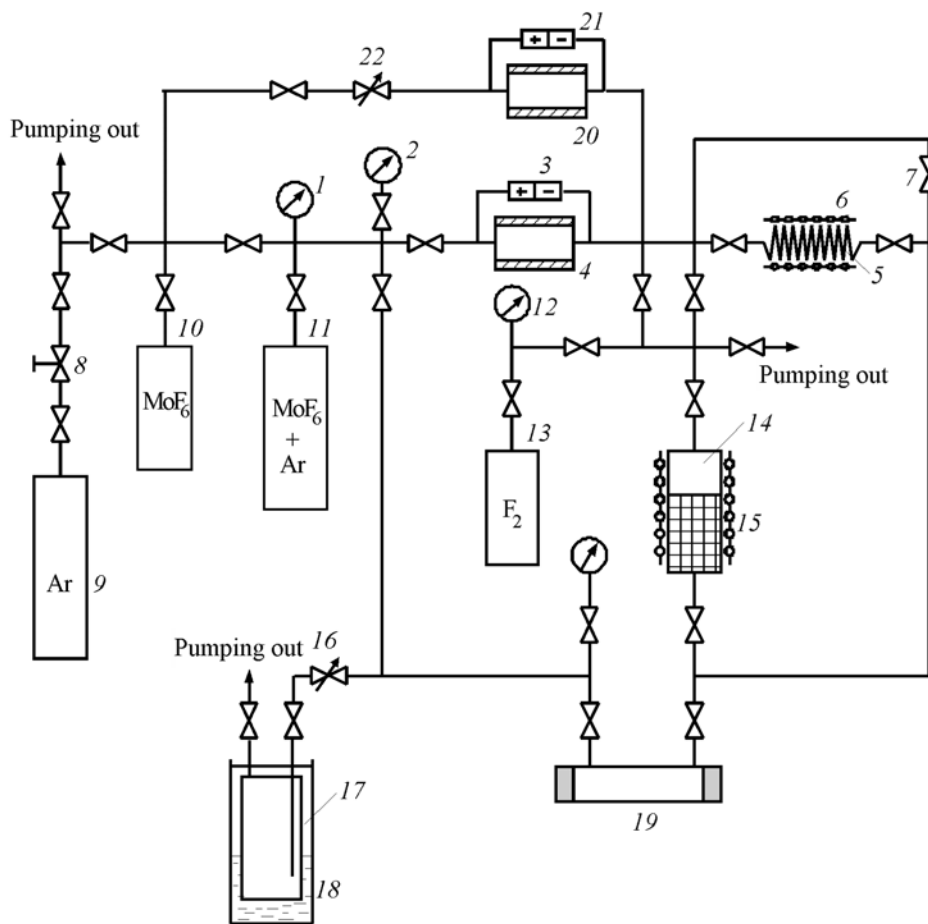|	
Composition of the sorbent	NaF
Strength, kgf/cm^2	8
Pore volume, cm^3/g	0.53
Surface (by the Brunauer, Emmett, and Teller method, m^2/g	2.0–2.5
Average pore diameter, nm	≈ 1000
Relative porosity, %	59.5

Fig. 1. Block diagram of the setup: 1–3, 12, and 21) "Sapfir-22" metering transducer; 4 and 20) gas resistor; 5) spiral manufactured from a nickel tube; 6 and 15) heater-thermostat; 7) bypass; 8) reducer; 9) cylinder with argon; 10) vessel with MoF_6; 11) vessel with a mixture (MoF_6 + Ar); 13) vessel with fluorine; 14) column with sodium fluoride; 16 and 22) throttle; 17) pumping-through trap; 18) Dewar vessel; 19) optical IR cell.

Experimental. *Experimental Setup.* To investigate the loss of MoF_6 in transport by service lines and to study its sorption on NaF we have created a setup shown in Fig. 1. All technological service lines of the setup (6×1 tubes) and closing devices — valves and throttles — are manufactured from 12Kh18N10T steel, and fluoroplastic is used as the sealant.

Before mounting the setup, we degreased the service lines and closing devices, washed them with nitric acid and water, and dried them. The interior surfaces of the units of the setup were treated with fluorine for removing traces of water and other contaminants. Taking account of the high reactivity of MoF_6 with respect to water, we performed fluorine treatment with molybdenum hexafluoride before each experiment. The pressure and the flow rate of the gaseous mixture were measured with "Sapfir"-type metering transducers. The temperature was monitored and measured with Chromel-Alumel thermocouples.

We used heat-insulated tube-type furnaces as heaters-thermostats; we placed a nickel tube twisted into a spiral in one furnace, modeling the route of the RAMUS plant, and a sorbent-containing column in the other. An Ar + MoF_6 gaseous mixture prepared in a stainless-steel vessel of volume 43.4 liters served as the working medium in the experiments. For this purpose we used high-purity argon and MoF_6 synthesized from the elements by the reaction

$$Mo + 3F_2 = MoF_6 . \tag{1}$$

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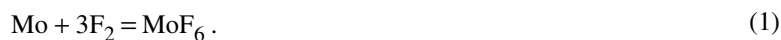
The purity of fluorine amounted to ~99%. The synthesized hexafluoride was purified by low-temperature vacuum distillation and was analyzed for the content of impurities using IR spectroscopy. When the gaseous mixture was prepared, the MoF_6 pressure in the vessel was measured with a "Sapfir"-22, model DA-2030, transducer, whereas for measuring the gaseous-mixture pressure in the setup, a "Sapfir"-22, model DA-2040, transducer was used.

The experimental bench allowed measurements in the temperature range 20–400°C for a flow rate of the gaseous mixture of 0.1 to 50 cm^3/sec. The flow rate of the gas was adjusted with a needle throttle by measuring the pressure drop on a precalibrated gas resistor representing a nickel capillary of diameter 0.5 mm and length 5 cm. The pressure drop on the capillary was measured using the Sapfir-22 DD-2420 transducer.

Argon was fed from a high-pressure cylinder. The argon flow rate was monitored by the pressure drop on the calibrated gas resistor manufactured from a half-ring-shaped stainless-steel tube with an inside diameter of 2 mm and a length of 23 cm. The pressure drop was measured with an AIR-20 M2-DD, model 400, pressure transducer.

For measuring the concentration of MoF_6 in the flow we used two optical cells: a single-pass one and a multipass one with optical-path lengths of 7.4 cm and 4.8 m respectively. The single-pass cell was manufactured from Kh18N10T stainless steel and was equipped with ArCl windows. Fluoroplastic served as the sealant.

All indicated materials were resistant to elementary fluorine at room temperature, which made it possible to treat the cell's interior surface with fluorine and to minimize the MoF_6 loss in the optical cell.

Measurement of the MoF_6 Loss in Transport by Nickel Service Lines. The MoF_6 loss in transport by nickel service lines was determined in a tube of diameter 8×1 mm and length 6 m, which modeled the RAMUS route. The tube was twisted into a spiral of diameter 6 cm and length 55 cm for reducing the linear dimensions and was placed in a thermostat. Before the experiments, the nickel tube was treated with nitric acid for decontamination of the interior surface, thoroughly washed with hot water and alcohol, and dried with hot air. We installed the tube prepared in this manner in the thermostat, connected it to the bench, evacuated it, and tested it for air-tightness. Then we degassed the tube by lengthy (for 5 h) pumping out by a liquid-nitrogen-cooled charcoal trap to a pressure of $<10^{-3}$ mm Hg, after which we passivated it by elementary fluorine. For this purpose the pumped-out tube was filled with fluorine to a pressure of 400 mm Hg, held in a fluorine atmosphere for 1 h, and then evacuated. Such a procedure was repeated several times with gradual increase in the temperature to 350°C. This produced a dense NiF_2 film on the nickel-tube surface. After passivation, the tube was filled with high-purity argon to a pressure of 1.1 atm. The remaining units of the setup were treated with elementary fluorine at room temperature and a fluorine pressure of 400 mm Hg.

A mixture of molybdenum hexafluoride and high-purity argon with a prescribed MoF_6 concentration was prepared in a vessel of volume 43.4 liters. Because of the presence of water in the argon, the mixture was held for several days for the reaction of hydrogen of a part of the MoF_6 to be completed and its concentration in the vessel with the gaseous mixture to be stabilized. The steady-state MoF_6 concentration determined from the intensity of the adsorption band at the frequency $\nu = 739.2$ cm^{-1} was $2 \cdot 10^{16}$ cm^{-3} at a mixture pressure of 760 mm Hg.

The order of the experiments on determining the MoF_6 loss in the nickel tube was as follows. The flow of the Ar + MoF_6 mixture with a rate of 0.5 cm^3/sec at atmospheric pressure was bypassed to the single-pass optical cell of the IR spectrometer around the nickel tube. We measured the transmission spectrum and determined the concentration of MoF_6 at the tube inlet from the transmittance at the frequency $\nu = 739.2$ cm^{-1}. Then we guided the flow to the cell through the tube and determined the concentration of MoF_6 at the tube outlet. Since the exchange time of the gas in the tube and the cell was ~6 min for a flow velocity of 0.5 cm^3/sec, the absorption spectra were recorded within 8 min after the establishment of the flow rate. The value of the MoF_6 loss in the tube was determined from the measured concentrations.

The procedure of measurement of the MoF_6 loss with increase in the tube temperature was as follows: with the aim of saving the gaseous mixture we decreased the flow through the tube to 0.2 cm^3/sec, heated it to the prescribed temperature, and thermostatted it. Then we increased the flow rate to 0.5 cm^3/sec again and determined, after 8 min, the concentration of MoF_6 at the tube outlet at the established temperature. The value of the MoF_6 loss was determined from the formula

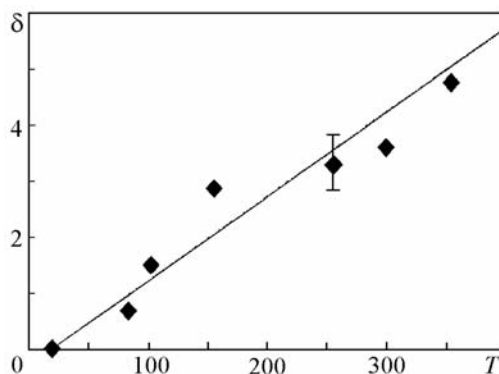$$\delta = \frac{n_{in} - n_{out}}{n_{in}} 100\% . \tag{2}$$

Fig. 2. Loss of MoF_6 vs. nickel-tube temperature. δ, %; T, $^{\circ}C$.

Figure 2 plots the MoF_6 loss as a function of the nickel-tube temperature.

The small monotonic increase in the loss with spiral temperature is attributable to the interaction of MoF_6 with the traces of crystallization water in NiF_2 formed during the fluorine passivation of the nickel spiral and sorbing water in blowing of the spiral with argon. It is common knowledge that in fluorination, crystallization water is difficult to remove from $NiF_2 \cdot nH_2O$ even at high temperatures, and the fluorination rate grows with temperature. In any case the expected MoF_6 loss in the service lines of the RAMUS reactor plant is small and no higher than ~0.5% at an operating temperature of 50 to $60^{\circ}C$.

Investigation of the MoF_6 Sorption on Granulated Sodium Fluoride. Infrared spectroscopy with a single-pass cell in the Fourier spectrometer makes it possible to measure MoF_6 concentrations in gas flows of no lower than ~10^{14} cm^{-3}, which is an order of magnitude higher than the content of [99]Mo in the actual system.

Nonetheless, pioneering experiments on the sorption of MoF_6 on granulated sodium fluoride were performed with a single-pass cell for its initial concentration of ~10^{16} cm^{-3} in the mixture with argon. The measurements were methodological in character and made it possible to obtain initial information on the trends of MoF_6 sorption on NaF at hexafluoride concentrations rather low now in the gaseous mixture.

Before the experiment, the sorbent underwent special treatment: to remove traces of water we evacuated the column for a long time (5 h) at $300^{\circ}C$ with a backing pump and then by a charcoal trap to a residual pressure of ~10^{-3} mm Hg. Thereafter we filled the column with fluorine to a pressure of 300 mm Hg, held it for 1 h, evacuated it, and allowed the fluorine to fill the column again. After holding for one hour, the system was evacuated to a residual pressure of 10^{-3} mm Hg.

The order of the experiment was as follows. The Ar + MoF_6 mixture prepared in advance was allowed to pass through a sorbent-containing column and was sent to the optical cell where the MoF_6 concentration in the gas flow was determined using an IR spectrometer. In the experiments, we used a column manufactured from nickel and with an inside diameter of 1.6 cm; the sorbent mass was 25 g; the bed height was 20 cm.

Figure 3a gives the absorption spectra of the Ar + MoF_6 mixture that has been allowed to pass by (curve 1) and through the NaF-containing column (remaining curves) at a temperature of $20^{\circ}C$. The spectra were recorded at time intervals indicated in the figure.

The measurements were performed at a flow rate of the mixture of 0.9 cm^3/sec and a pressure of 760 mm Hg in the optical cell. The concentration of MoF_6 at a mixture pressure of 760 mm Hg was $4.2 \cdot 10^{16}$ cm^{-3}. Within 6–7 min after the beginning of the blowing of the gaseous mixture through the sorbent-containing column, the MoF_6 concentration decreased below the sensitivity limit of the IR spectrometer with a single-pass cell ($1.5 \cdot 10^{14}$ cm^{-3}) and did not exceed this value till the end of the experiment. Over the period of measurement, the pressure in the vessel with the mixture decreased from 947 to 785 mm Hg. The mass of MoF_6 sorbed in the column is found from the relation
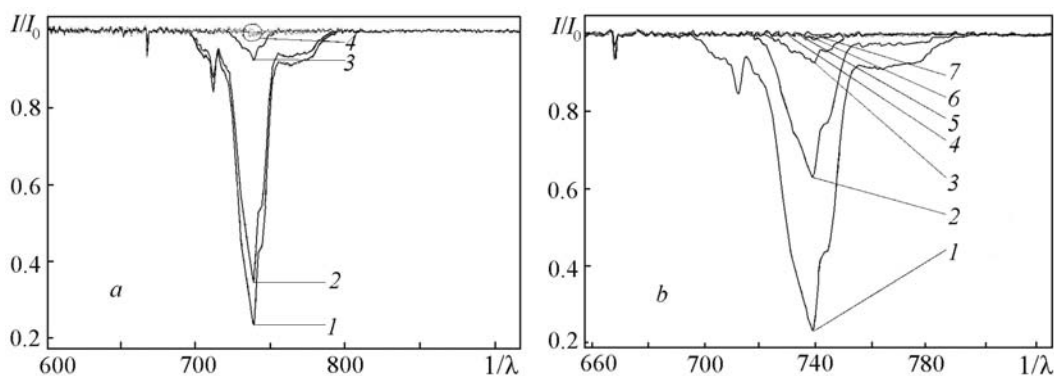
$$m = \frac{(P_1 - P_2)\, n_0 \mu V}{760 N_A}. \tag{3}$$

Fig. 3. Absorption spectra of the gaseous mixture Ar + MoF$_6$ at the column outlet vs. pumping-through time at $T = 20^{\circ}$C (a) and for different flow rates of the gas at $T = 50^{\circ}$C (b): a) 1) bypass; 2) 0 min; 3) 5 min; 4) 8 min; b) 1) bypass; 2) 11.4 cm^3/sec; 3) 11.4 cm^3/sec (pumping-through time 3 min); 4) 6.0 cm^3/sec; 5) 3.3 cm^3/sec; 6) 1.9 cm^3/sec; 7) 1 cm^3/sec.

Substituting the numerical values into (3), we obtain that 0.12 g of MoF$_6$ was absorbed in the column during the experiment.

Figure 3b gives the absorption spectra of the Ar + MoF$_6$ gaseous mixture that has been allowed to pass through the NaF-containing column at a temperature of 50°C for different flow rates of the gas. The concentration of MoF$_6$ at the column inlet is $2.1 \cdot 10^{16}$ at a pressure of 760 mm Hg. The remaining parameters of the experiment are the same as in the case of Fig. 3a. The pressure in the vessel with the mixture decreases from 934 to 875 mm Hg during the experiment.

From the given spectra, it is clear that the NaF-containing column with an inside diameter of 1.6 cm and a height of the poured sorbent bed of 20 cm is capable of ensuring efficient absorption of MoF$_6$ for flow rates of up to 6 cm^3/sec.

The breakthrough of MoF$_6$ in the column was observed for a flow rate of more than 11 cm^3/sec. To illustrate this effect Fig. 3b gives two spectra (2 and 3) for a flow rate of 11.4 cm^3/sec. Spectrum 3 corresponds to a time of pumping through the sorbent of 3 min. It is seen that the MoF$_6$ line appears in the gas flow, suggesting the beginning of the breakthrough of MoF$_6$. The breakthrough of MoF$_6$ grows with further pumping of the gaseous mixture through the sorbent (spectrum 2).

The computations performed from relation (3) show that ~0.03 g of MoF$_6$ has been sorbed in the column during the experiment, i.e., the dynamic capacitance of sodium fluoride (25 g) for a flow rate of 11.4 cm^3/sec and a concentration of MoF$_6$ at the column inlet of $2.1 \cdot 10^{16}$ cm^{-3} is ~0.03 g.

Measurements with a Multipass Cell. To investigate the sorption of MoF$_6$ at low concentrations in the gaseous mixture we used a multipass cell of the IR Fourier spectrometer and an NaF-containing column of larger volume. The use of the multipass cell required considerable increase in the mixture's flow rate. The reason was that the free volume of the multipass cell was equal to 2.4 liters. For a flow rate of the mixture of 1 cm^3/sec, the exchange time of the gas in the column was equal to 40 min. This was intolerably high because of possible corrosive interaction of MoF$_6$. The flow rate of the gas should have been increased to 20 cm^3/sec to obtain acceptable exchange time, of the order of 2 min. However, for such a high flow rate, the supply of the gaseous mixture in the vessel of 43.4 liters would last for 30 min of operation. To minimize this difficulty we obtained the required flow of the mixture by mixing the argon flow with a high rate and the mixture with a low rate but a high MoF$_6$ content.

The order of the experiments was as follows. The flow of the MoF$_6$ + Ar mixture prepared in advance was mixed with the argon flow and was pumped through the multipass cell by the shortest route. After fixed time intervals, we measured the concentration of MoF$_6$ in the flow. Once the steady-state concentration of MoF$_6$ had been reached, the gas flow was switched to the route passing through the column.

Figure 4 gives the time dependences of the content of MoF$_6$ in the flow at the outlet of the column filled with granulated sodium fluoride. The curve in Fig. 4a has been taken at a sorbent temperature of 20°C, whereas the
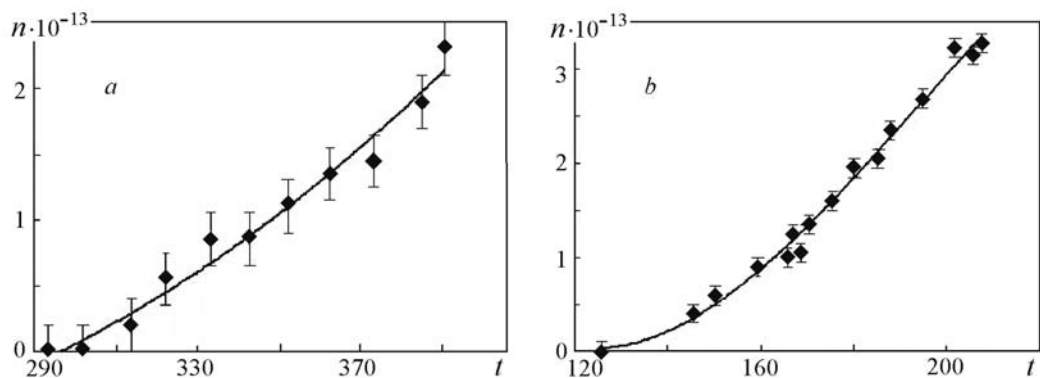
Fig. 4. Concentration of MoF_6 in the flow at the column outlet vs. time at $T = 20^oC$ (a) and $T = 50^oC$ (b). t, min; n, cm^{-3}.

curve in Fig. 4b has been taken at 50^oC. The experimental conditions are as follows: argon flow rate 35 cm^3/sec, mixture flow rate 0.45 cm^3/sec, and MoF_6 concentration in the starting mixture at a pressure of 760 mm Hg $3.3 \cdot 10^{16}$ cm^{-3}, which corresponds to a concentration of MoF_6 in the flow of $4.2 \cdot 10^{14}$ cm^{-3}.

If we take, as the "breakthrough threshold," a concentration of MoF_6 in the flow at the column outlet of $6 \cdot 10^{12}$ cm^{-3} (10% of the MoF_6 concentration in the experiments on the RAMUS plant), the sorptive capacity of sodium fluoride is 103.4 mg at $T = 20^oC$ and 46.6 mg at $T = 50^oC$. This is respectively 130 and 60 times larger than will be in the reactor experiment in one complete irradiation cycle.

Discussion of Results. The performed experiments show that granulated high-porosity sodium fluoride can be used for the extraction of MoF_6 from an inert-gas flow for concentrations of molybdenum hexafluoride of up to $2 \cdot 10^{12}$ cm^{-3}.

The sorptive capacity of NaF grows with MoF_6 concentration in the gaseous mixture and drops as the sorbent temperature increases from 20 to 50^oC. Deep extraction of MoF_6 from the gas flow occurs even for high flow rates of the mixture (11.4 cm^3/sec in the experiments with a sorbent mass of 25 g and 35 cm^3/sec in those with a mass of 112 g).

Let us evaluate the residence time of the mixture (τ) in the sorption column by the formula

$$\tau = \frac{v_{col} - v_{sorb}}{q}, \quad v_{sorb} = \frac{m}{\rho},$$

where $\rho = 1.05$ g/cm^3. The time of contact of the gas with the sorbent is equal to 2.7 sec in the first case and to 3.4 sec in the second case. It is only the external granular beds that operate efficiently, and a substantial increase in the sorbent capacity can be expected with decrease in the mixture's flow rate to 1 cm^3/sec.

As the temperature increases to 100^oC, the process of MoF_6 desorption becomes noticeable now. However, since the temperature where the sorbent-containing column is found will not be higher than 60^oC in the reactor experiments, it is quite justified to use NaF for extraction of MoF_6 in the RAMUS plant. The small amount of the absorbed MoF_6 with respect to the sorbent mass demonstrates that the process occurs mainly on the sorbent surface. To answer the question of the sorption mechanism we compare the specific characteristics of MoF_6 and SF_6 sorption by sodium fluoride. According to our measurements, the specific sorption (ε_{sp}) of SF_6 by sodium fluoride at a pressure of 0.76 mm Hg is less than $4 \cdot 10^{-8}$ $mole/m^2$. For MoF_6, this quantity is

$$\varepsilon_{sp} = \frac{0.12 \text{ g}}{210 \text{ g/mole} \cdot 25 \text{ g} \cdot 2.0 \text{ m}^2/\text{g}} = 1.1 \cdot 10^{-5} \text{ mole/m}^2.$$

As we see, in the case of physical adsorption of SF_6 the specific capacity is more than two orders of magnitude lower than that in the case of MoF_6 sorption. This, undoubtedly, is evidence in favor of the chemisorption mechanism.

Conclusions. Since the sorbent is in the zone of intense irradiation, we can expect improvement of its sorptive capacities due to the appearance of additional active chemisorption sites. In the literature [11], there are data on this effect in the γ-irradiation of alkaline-earth-metal fluorides.

NOTATION

I, radiation intensity, rel. units; m, mass, g; N_a, Avogadro number; n, concentration, cm^{-3}; P, pressure, mm Hg; q, flow rate of the gas, cm^3/sec; T, temperature, ^oC; t, time, min; V, volume, cm^3; v, volumes of the column and the sorbent, cm^3; δ, loss, %; ε, sorptive capacity, $mole/m^2$; λ, wavelength, cm; μ, molecular weight, g/mole; ν, radiation frequency, cm^{-1}; ρ, density, g/cm^3. Subscripts: sp, specific; col, column; sorb, sorbent; in, inlet; out, outlet; 1 and 2, initial and final values; 0, at a pressure of 760 mm Hg.

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