A description and schematic diagram of a rectification facility for separating uranium hexafluoride with different degrees of enrichment from process mixtures produced in separation operations at the Industrial Association Élektrokhimicheskii Zavod are presented. The results of operation of the facility over the period from the third quarter of 1971 to the third quarter of 1972 are reported. Different regimes for distilling mixtures, depending on the initial composition, are discussed. It is shown that there is promise in replacing hydrolysis of process mixtures of highly enriched uranium hexafluoride by rectification fractional distillation.

Efficient operation of equipment for separating uranium isotopes and meeting the stringent purity requirements for the product are impossible without removing impurities, mainly consisting of hydrogen fluoride, tungsten and molybdenum hexafluoride, volatile silicon, phosphorus, and chromium fluorides, and volatile organic substances (Freon-12, CH₂F₂), from the process cycle.

At the Industrial Association Élektrokhimicheskii Zavod, the impurities removed from the process line were reprocessed by the hydrometallurgical method. The high cost of reprocessing a unit of product, the multiple-stage nature of the process, and the long times required made it necessary to search for different methods of reprocessing. In addition, hydrolysis of uranium hexafluoride with high enrichment is dangerous because of the possibility of a nuclear reaction occurring, and the presence of a large quantity of fluorine-containing impurities increases uranium losses from the waste solutions.

Research work, previously performed at the Russian Science Center Kurchatov Institute showed that rectification is highly efficient for removing volatile fluoride compounds from uranium hexafluoride with the natural isotopic composition [1–4]. For this reason, it was useful to test separation of highly enriched uranium hexafluoride from real mixtures by means of rectification, i.e., to study the possibility of replacing hydrolysis of a mixture by rectification partial distillation.

To this end, in 1971, a prototype of a commercial rectification facility operating cyclically (see Fig. 1) was built and put into operation at the Élektrokhimicheskii Zavod. The facility was placed in a two-section box made of polymethyl methacrylate. Each section had its own ventilation exhaust system and a system for heating air up to 60°C. Air removed from the box was fed into the workshop’s ventilation system. A collector which accumulated a load of the initial mixture in the column still was located in one section of the box. Vapor (tank 1) and liquid (tank 2) phases were loaded. Weighing balances 8 automatically monitored the mass of the tanks. A cyclically operating packed rectification column, designed and built at the Russian Science Center Kurchatov Institute, was located in a different section of the box. The separation part of the column

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consists of two thermally insulated sections 4 filled with packing. The packing was held up by a false bottom. The liquid flowing down the top section was directed through dripping apparatus to the center of the packing surface in the bottom section. A typical tank was used as the still for column 3. It was placed inside a Dewar, into which liquid nitrogen was poured at the time the initial mixture was loaded. The column still was equipped with an electric heater whose power could be automatically regulated. The reflux condenser 5, placed above the packing in the top section, was cooled with liquid nitrogen. Part of the liquid condensing it (the reflux) flowed down through the dripping apparatus onto the packing of the top section of the column, and another part of the liquid was extracted through a control valve as distillate. The pressure was measured in the still and in front of the reflux condenser, and the temperature was measured at the top of the still and at six points along the packing part of the column and reflux condenser.

The distillate condensation collector consisted of two types of tanks 6, equipped with electric heaters, and settling tanks 7 connected in series with the first two tanks. All tanks of the condensation collector were placed in Dewars, into which liquid nitrogen was poured. The pressure and rate of extraction of the distillate were measured in the collector. In-line analysis of the composition of the initial mixture and distillate was performed continually with a mass spectrometer. Ten to twelve kilograms of the initial mixture were loaded for one fractional distillation operation.

The most laborious operation was found to be the fractional distillation of the content of tanks from the OK-0 purification cascade. A salient feature of the composition of these mixtures was a low concentration of uranium hexafluoride in the presence of a large quantity of volatile Freon-12 and hydrogen fluoride, whose concentration in individual cases reached 85 mass%. In the best case, conventional distillation of mixtures with this composition can yield uranium hexafluoride with hydrogen fluoride content corresponding to the composition of their azeotrope, i.e., about 30 mass% [5].

A special operating regime was developed for the setup to solve this problem. This regime made possible to remove volatile impurities without driving off the uranium hexafluoride. As the volatile impurities were extracted, the pressure in the column decreased and the content of (primarily) tungsten hexafluoride in the distillate increased. The pressure 3.5·10^2 kPa, the temperature of the reflux condenser 43°C, and the molar composition of the distillate (~74% tungsten hexafluoride) indicated that the tungsten hexafluoride was removed in the form of the azeotrope with hydrogen fluoride [3]. As the distillate was removed, the temperature of the still increased to 75–80°C and the pressure decreased to 2·10^2 kPa. Molybdenum hexafluoride appeared in the distillate, and the content of hydrogen fluoride increased to 50% by volume. The increase in the temperature of the still with the heat balance of the facility maintained constant showed that the content of volatile impurities in the still liquid decreased. The extraction of more distillate resulted in the appearance of uranium hexafluoride in the reflux condenser. Extraction of the first fraction, accumulating most of the impurities, was completed when the content of uranium hexafluoride in the distillate reached 0.1 vol. %. The composition of the first fraction (according to data from chem-