Reduction of uranium hexafluoride to tetrafluoride by using the hydrogen atoms

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Abstract. We consider the reduction of UF₆ to UF₄ by chemical reaction with hydrogen atoms originated in the powerful chemical generator. The principal design of such a chemical convertor is described. The results of the mathematical modeling of the thermodynamics and kinetics of the UF₆ to UF₄ reduction process are analyzed. The few options for the hydrogen atom generator design are proposed. A layout of the experimental setup with the chemical reactor is presented. The high efficiency together with the ability of the process scaling without loss of its efficiency makes this approach to the uranium hexafluoride depletion into tetrafluoride promising for its application in the industry.

1. Introduction
For the long period of nuclear materials production the more than 1.5 million tons of depleted uranium hexafluoride (DUH) has been accumulated in the world, with an annual increase of ~ 70 thousand tons. Storage of such DUH quantities is a serious and sophisticated problem, which encourages the search for the processes for its conversion into more safe for storage compounds (e.g., U₃O₈ or UF₄) being commercially demanded products [1,2].

One of well-known and investigated in Russia methods of the DUH conversion into more safe uranium tetrafluoride (DUT) is its reduction in hydrofluoric flame [1-3].

The reduction reaction of UF₆ + H₂ ⇌ UF₄ + 2HF due to its high activation energy requires the reaction temperature of 1500-2000 K [4]. It is assumed that the reagent can be heated in hydrofluoric flame: H₂ + F₂ ⇌ 2HF. The final products should be uranium tetrafluoride and dehydrated hydrogen fluoride. The main advantage of this method is the minimum energy expenses because the energy for heating the mixture released in chemical reaction.

The UF₆ reduction reaction with hydrogen becomes exothermic only after the UF₄ gas solidification, which can occur only at temperatures below 1600 K [4]. Thus, for successful conversion of UF₆ to UF₄ it is necessary to satisfy the contradictory requirements for the temperature to be sufficiently high for the reduction of UF₆ in gas phase, and, simultaneously, enough low for the further realization of UF₄ solidification. The solution of this contradiction can be contributed with the heterogeneous catalytic reduction of UF₆ on the reactor walls and/or on the surface of just formed solid UF₄ dust. The whole reduction process becomes in this case exothermic and self-sustaining [5,6].
It was proposed in [5, 7] to carry out the UF₆ reduction with pre-atomized hydrogen produced by a hydrogen atom generator, which makes the process more manageable and easy realized. It was proposed for that to use as the powerful chemical generator the system developed in the "NPO Energomash named after Academician VP Glushko" for CW chemical HF lasers (HFL), where the H atoms were the waste product. This system produces as much as 1 mole of hydrogen atoms per second and it was designed to work at high temperatures in corrosive environment (HF).

An important difference of this method for the uranium hexafluoride reduction is the absence in the initial mixture of molecular fluorine, which leads to fast competing $H + F_2 \rightarrow HF + F$ reaction of H atoms decay, and the presence of supersonic jet, preventing the heat propagation upstream and allowing such a way to work at high temperatures without perishing the mixing chamber [5].

Application of the hydrogen atom generator expands the abilities of the chemical reactor, provides high efficiency during process scaling-up, and open the possibility to study various schemes of the reduction process organizing.

National technologies of DUH reduction in hydrofluoric flame developed in JSC "ARRICT" and allowing to have DUT as the product are attractive because of the partial compensation of energy expenses for conversion process by the implementation of commercial products having a national economic significance. Indeed, in addition to dehydrated hydrogen fluoride as well as commodity products for microelectronics such as silicomethane, silicon tetrafluoride and others may be produced.

2. Scheme of the chemical reactor with an atomic hydrogen generator

The principal design of the reactor for UF₆ reduction with using the generator of atomic hydrogen where UF₆ supply into the nozzle arrays shown in figure 1 [6].

Thermochemical generators of hydrogen atoms having the efficiency required for an industrial application which are the main unit of the chemical reactor were created for CW HF chemical lasers (HFL). The typical generator is presented in figure 2 [7]. It consists of several basic units: array of burners, combustion chamber, nozzle array and reactor.

It operates as follows. In the combustion chamber the reaction of $H_2$ molecules with excess amount of $F_2$ takes place. The excessive weakly-bound $F_2$ molecules (dissociation energy of $F_2$ molecule on F atoms is only 37 kcal/mole) entirely dissociated at high temperature to fluorine atoms. These atoms are then tempered passing through the supersonic nozzle array and are in-flow mixed with molecular hydrogen. The subsequent fast chemical reaction $F + H_2 \rightarrow HF(v) + H$ results in H atoms formation which enter in the aimed chemical reaction with DUH.

![Figure 1](image1.png) 

**Figure 1.** The scheme of chemical converter [6].

The proposed technology for uranium hexafluoride to tetrafluoride reduction in the chemical reactor with an atomic hydrogen generator is close ideologically to the method for UF₆ to UF₄ reduction with hydrogen in the hydrofluoric flame. This technology combines the advantages of the UF₆ reduction method using molecular hydrogen in the hydrofluoric flame (having minimal energy
expenses) with removing the problems caused by endothermicity of the process at the stage preceding UF₄ condensation. This is due to the fact that both reactions of the UF₆ to UF₄ reduction with atomic hydrogen

$$\text{UF}_6 + H \Rightarrow \text{UF}_5 + \text{HF} + 69.5 \text{ kcal/mole} \text{ and } \text{UF}_5 + H \Rightarrow \text{UF}_4 + \text{HF} + 34.6 \text{ kcal/mole}$$

are highly exothermic and rather fast at temperatures of several hundred K. That is why no outer energy is needed to organize stationary burning and keep it stable at sufficiently low temperature.

3. Numerical modeling of reduction processes

The chemical reactor with hydrogen atoms generator allows organize various schemes of the reduction processes. Consider some of them.

3.1. Reduction with atomic hydrogen

Let us consider the work of a chemical reactor in the mode of uranium hexafluoride reduction with atomic hydrogen. A mixture of fluorine and hydrogen molecules is supplied to the combustor, while uranium hexafluoride with secondary hydrogen – to the nozzle unit. The mixture of weakly coupled molecules of fluorine and hydrogen molecules in excess of fluorine ($[\text{F}_2] / [\text{H}_2] = n > 1$) is ignited at the inlet to the combustion chamber. The reaction $\text{H}_2 + n\text{F}_2 \Rightarrow 2\text{HF} + 2(n-1)\text{F}$ is highly exothermic and the heat released is sufficient for dissociation of $\text{F}_2$ molecules excess. Combustion products arrive into the reaction zone through supersonic oxidizer nozzles where mix with secondary hydrogen supplied through interleave neighbor fuel nozzles. As a result of the reaction of $2(n-1)\text{F} + 2(n-1)\text{H}_2 \Rightarrow 2(n-1)\text{HF} + 2(n-1)\text{H}$, hydrogen atoms $\text{H}$ are formed and mixed with UF₆ in the reactor (and if necessary with a buffer gas - diluent). If the process is organized in such a manner that the UF₆ is reduced to uranium tetrafluoride according to formula: $2(n-1)\text{H} + (n-1)\text{UF}_6 \Rightarrow (n-1)\text{UF}_4 + 2(n-1)\text{HF}$, then for every mole of the recovered UF₆ 2 moles of hydrogen atoms are needed.

Kinetic calculations of the reduction process for this mode of reduction are made in [5, 6]. The ultimate goal of the one-dimensional calculations in [5] was to determine the degree of UF₆ reduction depending on the stoichiometry and temperature of the mixture, reagent flow rate, and the selectivity of the target product - UF₄. In the mathematical description of chemical reductions in the reactor the following simplifying assumptions were made in this study: the reagent mixing was assumed to be instant, the linear flow rate of the reactants - constant and equals 200 m / c; due to large areas of the reactor the heat-removing was neglected and the temperature of nozzle unit was assigned to constant value, while the temperature of the reagents was adjusted by the amount of the diluent gas $\text{N}_2$ added to the flow and the processes of diffusion and thermal conductivity were not taken into account. The coordinate along the axis of the reactor serves as an independent variable and the concentration and temperature at the reactor inlet as the initial conditions.
In figure 3, the results of calculations of the main characteristics for the reduction process are demonstrated [5] for the initial $[\text{UF}_6]_0$ concentration of $2\times10^{17}$ cm$^{-3}$. From this figure, which shows the concentration profiles of the reactants and products it can be seen that the initial reaction product is UF$_5$, its subsequent reduction and formation of solid particles occurs after the moment when almost no UF$_6$ left.

![Figure 3](image_url)

**Figure 3.** The concentrations of $(\text{UF}_5)_n$, $(\text{UF}_4)_5$, H, and UF$_6$, as the functions of the distance $X$ along the reactor. Flow velocity is $2\times10^4$ cm/sec, $[\text{UF}_6]_0 = 1\ (2\times10^{17}$ cm$^{-3})$, $[\text{H}]_0 = 3$, with no dilution with nitrogen [5].

The depth of UF$_6$ reduction, $\eta = 1 - [\text{UF}_6]/[\text{UF}_6]_0$ becomes full immediately after meeting the stoichiometric requirement $[\text{H}]_0 ([\text{F}]_0)/[\text{UF}_6]_0 = 2$, and only a ten-fold dilution of the mixture causes the need to increase the concentration of atoms in half (figure 4).

![Figure 4](image_url)

**Figure 4.** The yield of UF$_6$ conversion ($\eta = 1 - [\text{UF}_6]/[\text{UF}_6]_0$, vs the content of hydrogen atoms (1, 2) and fluorine (3) in the working mixtures; $[\text{UF}_6]_0 = 1\ (2\times10^{17}$ cm$^{-3}$). 1, 3 - without diluting the mixture (only HF$_0$), 2 - with dilution with nitrogen to $[\text{M}] = [\text{N}_2] + [\text{HF}]_0 = 10$ [5].

Numerical simulation of the UF$_6$ reduction by H atoms regarding the geometry of the nozzle array is considered in [6], figure5. The composition of the initial mixture is defined by conventional formula: $[\text{H}_2]^{(1)} + \alpha [\text{F}_2]^{(1)} + 2(\alpha - 1)K[\text{H}_2]^{(2)} + 2(\alpha - 1)\beta [\text{UF}_6]^{(2)}$. Symbols (1), (2) denote, the combustion chamber and the reactor zone respectively. At the description of chemical reactions and vibrational kinetics in the mixing zone a kinetic model of HF-laser was used supplemented by the reactions involving uranium components. The maximum concentration of atomic fluorine is achieved at $\alpha = [\text{F}_2] : [\text{H}_2] = 3$. In this case, the reduction of one molecule of UF$_6$ to UF$_4$ requires at least 2.5 molecules of H$_2$ and 1.5 molecule of F$_2$, forming five molecules of HF [6].

Figure 5 shows the results of calculations for the nozzle array spacing of 7 mm for the composition $\alpha = 3$, $K = 2.2$, $\beta = 0.3$. The molecular weights of the first and second flows are $\mu^{(1)} = 19.5$ and $\mu^{(2)} = 43.2$. 

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The ratio of the fuel (H$_2$) nozzle’s exit area to oxidizer (F$_2$) one is chosen to be equal to 2.5: 1. Calculation was performed for the pressure at nozzle’s exit plane $p_0 = 10$ Torr.

Figure 5. Distribution along the stream of the molar flows of components and of the temperature averaged over the cross-section. $P_0 = 10$ Torr and nozzle array spacing is $h = 7$ mm [6].

The total hydrogen flow rate per nozzle array unit area for this variant is 0.08 g / cm$^2$/ sec, the fluorine flow rate is 0.44 g / cm$^2$/ sec, and uranium hexafluoride flow rate is 1.58 g / cm$^2$/ sec. It is shown that a full reduction of hexafluoride and creation of the targeted product occurs in this case, at a length of about 15 cm, figure 5. A feature of this scheme is the need to realize the mixing of supersonic flows, one of which contains F atoms, and the second - H$_2$ and UF$_6$ mixture. The mixing is mainly due to the diffusion of the most lightweight component. At first, H$_2$ diffuses into the stream containing F atoms, and then the hydrogen atoms diffuse back into the flow of H$_2$ with UF$_6$, providing the reactions of hexafluoride and pentafluoride reduction [6].

3.2. Thermochemical reduction.

In this mode, fluorine molecules and a mixture of hydrogen and uranium hexafluoride is fed into combustor, (figure 2), i.e., only one chamber (combustion chamber) is used. Calculation of the equilibrium composition of a stoichiometric mixture of F$_2$ molecules with molecules of the hydrogen and uranium hexafluoride is made in [4]. The problem of finding the final thermodynamic equilibrium composition of the mixture of substances involved in the reactions is determined not by the type of chemical reactions, but only the composition of the initial mixture and the process of transition to the final state (at constant pressure, volume, temperature, etc.). This greatly simplifies the calculation task and allows to reduce it to a system of ordinary nonlinear equations.

Dependences of thermodynamic equilibrium concentrations and their temperature on the initial mixture composition concentrations were obtained at solving the system of equations describing the equilibrium state in the system UF$_6$ - H$_2$ (H) - F$_2$ (F) - HF. The initial mixture is represented in a general form: H$_2$+($\alpha+\beta$)F$_2$+(1-$\alpha$)UF$_6$. One can obtain any composition of interest by varying the coefficients $\alpha$ and $\beta$. A complete partial pressure and the temperature of the individual components in thermodynamically equilibrium mixture were obtained depending on the composition of the initial mixture of H$_2$+($\alpha+\beta$)F$_2$+(1-$\alpha$)UF$_6$ at the initial temperature $T_0 = 373$ K and $p_0= 1.5$ atm.

In figure 6, the calculated thermodynamic equilibrium concentration ratio for UF$_5$ / UF$_4$ with taking into account gas and condensed phases for the starting composition H$_2$+($\alpha+\beta$)F$_2$+(1-$\alpha$)UF$_6$ at $T_0 = 373$ K and $p_0 = 1$... 10 atm is shown at a linear scale.
Figure 6. UF₅ / UF₄ concentration ratio in the thermodynamically equilibrium mixture, taking into account gas and condensed phases coexistence, for the initial composition H₂+αF₂+ (1−α)UF₆ at \( T_0 = 373 \) K and \( p_0 = 1 \ldots 10 \) atm. The values of the pressure are shown above the appropriate curves, [4].

The figure clearly shows that the content of UF₅ in the equilibrium mixture is small and varies within the range of \( 3 \div 12\% \) of the total UF₄ depending on \( \alpha \) and pressure \( p_0 \). A sharp curve bend of the dependency \( [\text{UF}_5]/[\text{UF}_4] \) on \( \alpha \) is due to the start of the formation of a condensed phase.

The formation of the condensed phase, on the one hand, removes the condensed component of the individual component concentration from the process of formation of an equilibrium gas composition; on the other hand, this is accompanied by the release of additional heat to the mixture due to the heat of gas-liquid phase transition. The formation of the liquid phase causes a drop of the UF₄ concentration in the gas phase, so the temperature dramatically slows down its decline due to heat release during the phase transition. Therefore, despite the temperature decreasing at \( \alpha \) reduction, the relative concentration of UF₅ starts to fall.

3.3. Combined reduction (with molecular and atomic hydrogen together).

In this mode, the uranium hexafluoride is supplied to the first zone (combustion chamber) together with fluorine and hydrogen, the combustion products flow into the second zone (reactor) through the oxidizer nozzles of the generator, and the hydrogen is fed there through the fuel nozzles, figure 2. This method provides a two-stage reduction - the thermal conversion in the first zone and an additional reduction by H atoms in the second zone. Numerical simulation of the reduction process for two different geometries of the nozzle array is given in [6]. The fields of pressure and temperature in the area of mixing the oxidizer and the fuel streams, as well as the distribution along the stream of the component’s mole flow rates and of the temperature averaged over the cross section, are obtained, figure 7.
Figure 7. Distribution of the mole flow rates of components and the temperature averaged over the cross section of reactor. \( P_0 = 10 \text{ Torr} \). The nozzle array spacing is supposed to be 14 mm (left) and 7 mm (right) [6].

Figure 7(left) displays the downward distributions of the mole flow rates of components and of the static temperature averaged over cross-section with taking into account the heat capacities of components. As seen, the uranium pentafluoride UF\(_5\) near the outlet of the nozzle array rapidly and almost completely dimerizes increasing the average temperature of the flow by \(~130\text{K}\). At a distance of a few millimeters from the nozzle array the processes of uranium fluoride reduction come into effect, as evidenced by the decrease of H concentration. The decrease of the UF\(_6\) concentration by two orders of magnitude and the creation of the target product UF\(_4\) and \((\text{UF}_4)^2\) almost completely ends at distances of \(~20\text{ cm}\) from the nozzle array exit plane. The average static pressure increases rather quickly with temperature increasing: at the distance of 1 cm to \(~20\text{ Torr}\), at 6 cm to 32 Torr, and at 30 cm to 83 Torr. The length of the reduction for this kinetics is of the order of 15-20 cm.

Calculation was also made at halving the mixing step of the nozzle array \( h = 7 \text{ mm} \), the results are shown in figure 7 (right). At a more intense mixing the reduction zone shortens, in this case - to \(~3.5\text{ cm}\), that corresponds to the length of hydrogen diffusion into the oxidizer stream.

The pressure at the nozzle unit exit was also varied. The higher is the pressure, the more easy to organize the pumping system. Fourfold increasing of the pressure at the nozzle unit exit – up to \( P_0 = 40 \text{ Torr} \) at an array spacing of 7 mm, naturally, slows the mixing of the secondary oxidizer and fuel flows, and lengthens the reduction zone to \(~17\text{ cm}\). This pressure increasing (and hence, flow rate) leads to an increase of the pressure at the end of the reduction zone to \(~220\text{ Torr}\), that makes it possible to relatively easy to perfume the exhaust of the combustion products at atmospheric pressure [6]. Performed calculations show the fundamental possibility of UF\(_6\) to UF\(_4\) reduction using atomic hydrogen generators of supersonic cw chemical lasers of high performance.

However, too much variation in literature for values of the constants of elementary chemical processes, in particular, of the reactions \( H + \text{UF}_6 \) and \( H + \text{UF}_5 \), creates certain difficulties in formulating the requirements for the design of a supersonic nozzle unit for variants of the reactor for DUH reduction using atomic hydrogen. For more accurate calculations it is necessary to clarify the experimental rate constants of certain chemical reactions (direct and inverse).

4. Experimental setup with a hydrogen atoms generator.

Figure 8 schematically illustrates the layout of the experimental chemical reactor with a hydrogen atoms generator. There are a few basic units of the experimental setup: gas supply system (gases \( \text{H}_2, \text{F}_2, \text{N}_2 \), etc.), system of preparation of \( \text{UF}_6 \), a chemical reactor, diagnostics, device of particulate collection \( \text{UF}_4 \) (cyclone unit, metal ceramic filters), vacuum receiver, ventilation systems and special ventilation. The entire installation is inside gas safety container.
Figure 8. A general view of the experimental setup with the hydrogen atoms generator. 1 - system of gas supply, 2 - cylinders for UF₆, 3 - chemical reactor, 4 - vacuum receiver, 5 - cryogenic trap, 6,7 - columns with NaF and filters, 8 - vacuum pump, 9 - hydrogen afterburner, 10 - special ventilation system, 11 - ventilation system, 12 - Fourier spectrometer, 13 - gas safety container, 14 - cyclone unit for UF₆.

The system of UF₆ preparation should provide the necessary flow rate of UF₆. The diagnostic system includes sensors of temperature, pressure, and flow rate; optical measurements (Fourier spectrometer) are needed to determine the concentrations and temperature of chemical mixture reagents. The vacuum receiver provides the coordination of the gas-dynamic channel of the chemical reactor. The system is equipped with a special ventilation set of filters and cryogenic traps to neutralize the exhaust.

The above-considered options of using the hydrogen atoms generator designed by the «NPO Energomash», allow a defluorination of uranium hexafluoride to the reduced consumption of up to 5 g / cm² / s, which is quite enough to clarify the calculation of experimental models and processing of the setup components. With this performance the setup has quite acceptable dimensions, figure 8. Theoretically, the continuous operation of the hydrogen atoms generator with its area of 400 cm² is capable of reducing with a capacity of up to 7 tons of depleted uranium hexafluoride per hour, provided that the gasification system and supply of the required flow rate of DUH exist. [6]

Mathematical modeling of thermodynamics and kinetics of the UF₆ to UF₄ reduction demonstrates the feasibility of the proposed method at quite reasonable parameters of the experimental device. Nevertheless, there is a certain variation in the data for some of the reaction rate constants, their refinement could lead to adjustments in the working parameters of the experimental chemical reactor.

5. Discussion
To be self-sustaining and providing a high yield the process of the UF₆ to UF₄ reduction must be exothermic. The analysis performed shows that the reaction of UF₆ + F₂ + H₂ → UF₄ + HF at any ratio of reagent contents may be exothermic only provided the UF₄ solidification during the reaction course. But in accordance with the general considerations and numerical calculation presented in this paper,
the UF₄ condensation occurs only if the reaction temperature is less than some specified value T *. Therefore, in principle, there are three possible approaches to the UF₆ to UF₄ reduction when using F₂ + H₂ reaction.

1. The reduction reaction in a mixture of UF₆ + F₂ + H₂, in a burner when, either by selecting the relative concentrations of the reactants, or by diluting the active mixture with an inert gas, the combustion temperature is less than T *. In this case it is possible to arrange a self-sustaining combustion in the form of a “smoky” torch, therein the smoke is particles of UF₄, which are carried away by the flow and can land on the cooled panels downstream. Wherein, of course, there are no difficulties in the transition to large-scale reactor. Unfortunately, the level of knowledge of the rate constants for chemical reactions occurring in the reactor does not allow us to be sure that this mode can be implemented because a relatively low temperature of the reaction means small rates of elementary processes. Furthermore, in this mode, it is difficult to maintain a stable combustion for a long time due to the nonlinear dependence of the reaction rates on the rate of UF₄ condensation. Experience of managing the combustion of hydrocarbons on conditions with heavy soot supports this conclusion.

2. Carrying out the reaction UF₆ + F₂ + H₂ in a burner at temperatures higher than T *, without any dilution of the active mixture with inert gas, but introducing into the reaction zone the panels cooled from inside to the temperature less than T *. Then, the product will condense on panels surfaces and the reaction rate will be high, including quite probable catalytic effect of surfaces coated with UF₄. In this regard, of interest are the experiments that have been already performed in JSC "ARRICT" as well as those that can be performed in the future. The disadvantages should include the complexity of the introduction of panels into the reaction zone, especially at large flows of reagents.

3. Using the chemical generator of hydrogen atoms. Since the UF₆ + H → UF₄ + HF reaction is exothermic even in the absence of UF₄ condensation, it will occur under all conditions and at any scale of the burner. Positive is the fact that in this case the product is formed downstream and beyond the reaction zone. Negative factor in this approach is that the consumption/ of the reagents and, above all, fluorine per one molecule of the converted uranium fluoride is higher than in the first two approaches, because of the additional fluorine should be spent in a hydrogen atoms chemical generator of. This approach is the most technically complex, but its complexity is mainly associated with the generator of atoms, and it should be taken into account that such generators producing flows of atoms required for a full-scale setup already exist.

In general, one should take into account that these approaches are not entirely independent. For example, in the frames of the third approach, one can try to reduce the initial concentration of hydrogen atoms by adding a certain amount of molecular hydrogen and fluorine into the mixer, thereby reducing the necessary flow of fluorine.

6. Conclusion
Taking into account the extensive experience of this paper authors in work with fluorine, hydrogen, fluorides and uranium, one should be argued that the problem of the UF₆ to UF₄ reduction by using fluorine with hydrogen at high reactant flows will be successfully solved. When using the more technically complex and more costly approach with a chemical generator of hydrogen atoms, the combined schemes can be applied simultaneously to minimize the expense of fluorine. The proposed method for reduction of uranium hexafluoride by use of a generator of hydrogen atoms is protected by RF patent №2204529 from 20.05.2003, [7].

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