

# Physicochemical Principles of Preparation of U(VI) Carbonate Solutions for Extraction Reprocessing in the Carbox Process

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Received December 13, 2016

**Abstract**—Physicochemical principles of preparation of U(VI) carbonate solutions in the step of oxidative dissolution of U<sub>3</sub>O<sub>8</sub> and UO<sub>2</sub> in the Carbox process are considered. Carbonate solutions with the U(VI) concentration higher than 100 g L<sup>-1</sup>, suitable for subsequent final purification of uranium by extraction, can be prepared under the conditions of formation of U(VI) carbonate–peroxide complexes in the course of dissolution with prevention of hydrolysis of U(VI) compounds. The behavior of impurities simulating some fission products in the course of oxidative dissolution was studied, and the decontamination factors of U(VI) from the chosen simulated fission products were determined.

**Keywords:** spent nuclear fuel, uranium dioxide, triuranium octoxide, oxidative dissolution, uranium(VI) peroxide–carbonate complexes, extraction from carbonate solutions

**DOI:** 10.1134/S106636221704004X

Today, the main industrial procedure for spent nuclear fuel (SNF) reprocessing is the Purex process. Its flowsheet was subjected to improvements for many years. In the Purex process, SNF is dissolved in HNO<sub>3</sub>, and final purification of U and Pu is performed by extraction with a 20–30% solution of tri-*n*-butyl phosphate (TBP) in a hydrocarbon diluent (HCD) [1].

The drawbacks of this process are well known and have been described in [2]. The results of studies on improvement of the Purex process or on development of alternative procedures for SNF reprocessing are described in [3, 4]. The main goals of these studies are improving the production safety (fire and explosion safety) and reducing the radioactive waste (RW) volume. One of the drawbacks of the Purex process, increased fire and explosion hazard caused by contact of HNO<sub>3</sub> of relatively high concentration with organic substances (extractant, HCD), is difficult to overcome within the framework of the existing (and widely used) sequence of operations. This fact became one of the main factors stimulating active search for alternative methods of SNF reprocessing, among which much attention is paid to dry methods: fluoride gas, pyrometallurgical, and pyroelectrochemical processes [4].

Among wet chemical methods of SNF reprocessing, we should mention improved versions of the Purex process and alternative methods of SNF reprocessing in alkaline carbonate media [3].

In the late 1990s, Japanese researchers formulated their own concept of SNF reprocessing, based on the sequence of the following operations: electrochemical dissolution of uranium SNF in an aqueous Na<sub>2</sub>CO<sub>3</sub>–NaHCO<sub>3</sub> solution, precipitation of cesium with sodium tetraphenylborate (STPB), separation of insoluble carbonates (together with the Cs precipitate) of some fission products (FPs), precipitation of U, Pu, Np, and Zr hydroxides from the clarified carbonate solutions, precipitation of Tc from the residual solution with tetraphenylphosphonium (TePP) chloride, and further ion-exchange purification of U and Pu after dissolving the hydroxide precipitate [5–9]. The suggested flowsheet proved to be efficient, and sufficiently pure uranium concentrate suitable for further processing was obtained.

The carbonate flowsheet was also studied at the Los Alamos National Laboratory (the United States) [10–15]. The principal process scheme suggested by the US scientists consists of the following operations: SNF

voloxidation, oxidative (in the presence of  $\text{H}_2\text{O}_2$ ) dissolution of uranium oxides in carbonate solutions, and partitioning of U and FPs by precipitation. The separation of U and Pu is based on precipitation of compounds of type  $\text{Na}_3\text{Pu}_2(\text{O}_2)_2(\text{CO}_3)_6 \cdot 12\text{H}_2\text{O}$ , Pu(IV) peroxide-carbonate complexes, from a carbonate solution in the presence of excess  $\text{H}_2\text{O}_2$  (there is certain analogy with final purification of uranium by peroxide precipitation). In the process, Np(V) coprecipitates with Pu(IV).

Korean researchers also took active part in the development of the carbonate-peroxide process for SNF reprocessing [16–20]. According to the scheme that they suggested, SNF was dissolved in an alkaline carbonate solution at pH 11–12 in the presence of  $\text{H}_2\text{O}_2$ . After the separation of the precipitate of insoluble FPs, technetium (with TePP chloride) and cesium (with STPB) were successively precipitated from the carbonate solution obtained, after which the solution was acidified with nitric acid to pH 3–5. The latter operation resulted in precipitation of uranyl peroxide (full analogy with final purification of uranium by peroxide precipitation).

The above-considered carbonate flowsheets for SNF reprocessing include precipitation operations using relatively expensive chemicals. Precipitation methods do not ensure the decontamination factors of U from FPs of the order of  $10^6$  (as in the Purex process). Furthermore, replacement of the purification by extraction (allowing, in contrast to precipitation methods, the decontamination factor to be increased by increasing the number of extraction steps and also the purification process to be performed repeatedly) with purification by precipitation will lead to significant changes in the primary extraction flowsheets.

The concept of the Carbex process, suggested by researchers of the Mendeleev University of Chemical Technology of Russia in 2008 [21], also involves the use of carbonate media for SNF reprocessing, but, in contrast to the above-considered precipitation flowsheets, involves final purification of both uranium and plutonium by extraction from carbonate solutions using organic solutions of quaternary ammonium carbonates (QACs) as solvents.

In the sequence of operations, the Carbex process flowsheet repeats that of the Purex process, including SNF voloxidation, oxidative dissolution of the voloxidized fuel in carbonate solution, extraction of U(VI) and Pu(IV) or Pu(VI) from the carbonate solution

(analog of the first extraction cycle of the Purex process), solid-phase stripping of U(VI) and Pu(VI) from the loaded solvent products, separation of the carbonate precipitates from the strip and their dissolution in a carbonate or carbonate-peroxide solution, final purification of U(VI) to remove FPs by extraction from carbonate solution, followed by solid-phase stripping of the U(VI) carbonate compounds (analog of the uranium branch of the Purex process), and final purification of Pu(VI) by extraction from carbonate solution, followed by solid-phase stripping of Pu(VI) carbonate compounds (analog of the plutonium branch of the Purex process). Along with separate final purification of U and Pu, it is possible to perform joint final purification of U(VI) and Pu(VI) by extraction from carbonate solutions, isolation of U(VI) and/or Pu(VI) carbonate precipitates, and their subsequent calcination in a reducing atmosphere to dioxides to obtain powders suitable for the production of ceramic nuclear fuel [22].

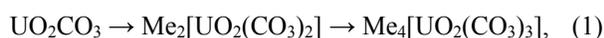
As in the other flowsheets, the suggested head end of the Carbex process is voloxidation, i.e., high-temperature oxidation of SNF with atmospheric oxygen and preparation of the fuel composition for further reprocessing. Voloxidation involves removal of gaseous and highly volatile FPs and oxidation of  $\text{UO}_2$  to  $\text{U}_3\text{O}_8$ , accompanied by disintegration of oxide crystals, which accelerates the uranium dissolution [23].

It is suggested that the voloxidation in the Carbex process be performed in the presence of alkali metal carbonates at 450–900°C to stabilize uranium in the hexavalent state in the form of alkali metal diuranates and monouranates [24–26]. The next step of reprocessing of oxidized SNF is preparation of stable carbonate solutions of U and Pu, suitable for performing subsequent extraction. In the above-cited studies using precipitation methods for processing carbonate solutions, the problem of preparing concentrated U(VI) carbonate solutions was not formulated explicitly, and the major attention was paid to decontamination from FPs via precipitation from carbonate solutions. Indeed, in Asano and Tomiyasu's paper [5], which is one of the first papers concerning preparation of SNF carbonate solutions, the authors performed anodic dissolution of  $\text{UO}_2$  in an  $(\text{NH}_4)_2\text{CO}_3$  solution. They obtained carbonate solutions with a uranium concentration of up to  $15 \text{ g L}^{-1}$ .

Apparently, efficient implementation of the extraction process requires preparation of carbonate solutions

with sufficiently high uranium concentration. Therefore, we considered in this study the physicochemical principles of preparation of U(VI) carbonate solutions suitable for the subsequent extraction processing.

Kim et al. [16] performed oxidative dissolution of SNF in an  $\text{Na}_2\text{CO}_3$  solution. The uranium concentration in the solution was  $83 \text{ g L}^{-1}$ . The time in which the dissolution equilibrium was attained (from 1–2 to 25–30 min) depended on the  $\text{H}_2\text{O}_2$  concentration (1–4 M) and initial form of the oxide ( $\text{U}_3\text{O}_8$  or  $\text{UO}_2$ ).  $\text{U}_3\text{O}_8$  dissolved appreciably more slowly than  $\text{UO}_2$ . For a long time, the U(VI) concentration in carbonate solutions of  $83 \text{ g L}^{-1}$  was the maximum reached. It should be noted that dissolution of U(VI) compounds in carbonate solutions without  $\text{H}_2\text{O}_2$  is accompanied by formation of the complex compound  $\text{Na}_4[\text{UO}_2(\text{CO}_3)_3]$  whose solubility in aqueous  $\text{Na}_2\text{CO}_3$  solutions does not exceed  $30\text{--}40 \text{ g L}^{-1}$  counting on U(VI). For example, when using atmospheric oxygen as oxidant, the following transformations of uranium carbonate compounds take place in carbonate solutions [27]:



where  $\text{Me} = \text{Na}^+, \text{NH}_4^+, \text{or } \text{K}^+$ . The use of  $\text{H}_2\text{O}_2$  leads to the formation of a mixed complex of the composition  $\text{Me}_4[\text{UO}_2(\text{O}_2)(\text{CO}_3)_2]$  [10, 14, 19, 27], which is considerably more soluble than  $\text{Me}_4[\text{UO}_2(\text{CO}_3)_3]$ .

On the other hand, the U(VI) concentrations reached in the course of dissolution of the fuel in  $\text{HNO}_3$  (Purex process) exceed  $300 \text{ g L}^{-1}$ . One of the main requirements to SNF reprocessing procedure that would be competitive with the Purex process is formation of solutions with comparable U(VI) concentration.

Because the main uranium form in the voloxidized fuel is  $\text{U}_3\text{O}_8$  (with possible presence of unchanged  $\text{UO}_2$ ), the overwhelming majority of experiments on uranium dissolution in carbonate solutions were performed using  $\text{U}_3\text{O}_8$  as starting compound. Dissolution of  $\text{UO}_2$  was also studied. The use of  $\text{H}_2\text{O}_2$  as an oxidant was substantiated in [10–20]. As noted in [28], the choice of  $\text{H}_2\text{O}_2$  is governed by its high oxidizing power in carbonate solutions, relatively high rate of U(IV) oxidation, and capability of the peroxy group to act as a ligand with the formation of mixed uranyl carbonate–peroxide complexes, increasing the solubility of U(VI) in carbonate solutions. Excess  $\text{H}_2\text{O}_2$  rapidly and readily decomposes in carbonate solutions with the formation of water and oxygen released into the gas

phase, after which the carbonate solution no longer exhibits oxidizing activity toward the organic phase and equipment materials.

An increase in the  $\text{H}_2\text{O}_2$  concentration accelerates the uranium dissolution [13]. Similar effect is observed with increasing alkali metal carbonate concentration and temperature [15, 29].

As indicated in [30], the time of the  $\text{H}_2\text{O}_2$  decomposition in carbonate solutions is 15–20 min. The advantage of the removal of the free oxidant is leveled off by the need for maintaining its concentration on a constant level in the course of leaching. An increase in the uranium concentration leads to the formation of the polynuclear complex  $\text{Me}_6[(\text{UO}_2)_2(\text{O}_2)(\text{CO}_3)_4]$  [10, 12, 29]. The U(VI) concentrations that can be reached in the course of oxidative dissolution of  $\text{UO}_2$  or  $\text{U}_3\text{O}_8$  in the presence of  $\text{H}_2\text{O}_2$  in carbonate solution exceed  $80 \text{ g L}^{-1}$  in terms of the metal.

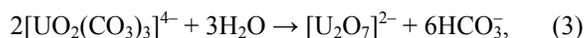
Dissolution in carbonate–peroxide solutions is considerably intensified by ultrasonic treatment of the reaction medium. For example, in straight dissolution of weighed portions of  $\text{U}_3\text{O}_8$  powder (mean particle size  $8\text{--}9 \mu\text{m}$ ,  $S : L = 1 : 5$ ,  $25^\circ\text{C}$ ), the time of complete dissolution is several hours, and the maximal uranium concentration in the solution reaches  $60\text{--}90 \text{ g L}^{-1}$  [30], whereas direct ultrasonic treatment decreases the dissolution time to 30–45 min. The dissolution rate constant in Jander's equation describing the process kinetics increased by 1–2 orders of magnitude, and the maximal U(VI) concentration in such solution was  $\sim 200 \text{ g L}^{-1}$  [29]. A study by derivative electronic spectroscopy has shown that uranium occurs in the solution mainly in the form of nondissociated mononuclear and polynuclear peroxide–carbonate complexes [31, 32].

The kinetic data obtained in [29] demonstrated the possibility of the occurrence of parallel processes in solutions: oxidative dissolution of  $\text{U}_3\text{O}_8$  and hydrolysis of the compounds formed. The hydrolysis decreases the dissolution rate, and secondary precipitates are formed. The kinetic curves of the dissolution pass through a maximum. The hydrolysis mechanism differs depending on the medium in which the process is performed:

in alkaline solutions,



in neutral solutions,



Decontamination factors of U(VI) from FP impurities in the step of oxidative dissolution of simulated uranium SNF (preliminary conversion of  $\text{UO}_2$  to  $\text{UO}_4$ )

Element	Content of U and FP impurities			$K_{\text{dec}}$
	pellet, $\text{mg g}^{-1} \text{U}_3\text{O}_8$	powder, $\text{mg g}^{-1} \text{U}_3\text{O}_8$	solution, $\text{mg L}^{-1}$	
U	800	800	74 880	–
Mo	4.6000	4.7333	148	2.9
Zr	5.7500	5.5333	16.1	33.4
Y	0.8400	0.6400	0.1	786
Ba	4.9500	6.8667	19.9	23.3
Sr	0.6350	0.6100	2.68	22.2
Al	0.2550	0.0353	0.96	24.9
Ca	0.0100	0.0470	4.52	0.2
Cs	0.0011	0.0023	0.21	0.5
Cu	0.0100	0.0093	0.19	4.9
Sn	0.2900	0.4133	0.1	271.4
La	3.8500	3.6333	0.1	3604
Nd	3.3500	5.8333	0.1	3136
Sm	1.0200	1.2667	0.1	955
Ce	3.2000	3.0333	0.1	2995
Cr	0.0100	0.0100	0.1	9.4
Mn	1.0900	1.1333	0.1	1020
Pd	0.2700	0.2640	0.1	253
Sb	0.0100	0.0100	0.1	9.4

under the conditions of formation of gaseous  $\text{CO}_2$ ,



Another way to prepare concentrated U(VI) carbonate solutions is dissolution of  $\text{UO}_2$  preliminarily converted to peroxide  $\text{UO}_4$ , e.g., in a solution containing 0.5–1.0 M  $\text{H}_2\text{O}_2$  + 0.05 M  $\text{H}_2\text{SO}_4$ . Under common conditions (room temperature, stirring), the conversion of solid oxide  $\text{UO}_2$  to  $\text{UO}_4$  powder, also solid, occurs slowly, in more than 8 h, whereas the converted product readily and rapidly dissolves in a 1 M aqueous  $\text{Na}_2\text{CO}_3$  solution. The conversion of  $\text{UO}_2$  to  $\text{UO}_4$  is considerably accelerated by ultrasonic treatment of the slurry, with the complete conversion time becoming as short as 45–60 min. Without ultrasonic treatment, a solution containing 54.5 g  $\text{L}^{-1}$  U(VI) in the form of a peroxide–carbonate complex was obtained in 2 h at room temperature. Dissolution of the same converted peroxide product under ultrasonic treatment allowed the U(VI) concentration reached in 60 min to be increased to 76.6 g  $\text{L}^{-1}$ .

Theoretical calculation of the initial U(VI) concentration required for performing final purification by extraction from carbonate solution showed that it should be in the range from 40 to 160 g  $\text{L}^{-1}$  [29]. The above-considered data on dissolution of uranium oxides in carbonate solutions in the presence of  $\text{H}_2\text{O}_2$  show that it is quite possible to reach such parameters and ensure efficient purification of U(VI) to remove FP impurities by extraction from carbonate solutions.

Oxidative dissolution of uranium SNF in carbonate solutions is accompanied by partial FP transfer into the aqueous phase. Some impurities, e.g., Cs and Mo(VI) compounds, virtually completely dissolve in carbonate solutions, and some other impurities dissolve partially. A part of impurities are separated from the dissolved U(VI) compounds in the step of preparation of the initial solutions for extraction purification. The efficiency of the uranium decontamination from FPs in the process as a whole, performed in carbonate media, is thus improved. The decontamination factors of U(VI) from some FP impurities, reached in oxidative dissolution of simulated uranium SNF in carbonate solutions, are given in the table. Simulated uranium SNF in the form of both a pellet and a powder was converted to a peroxy compound by treatment with an aqueous solution of 0.5 M  $\text{H}_2\text{O}_2$  + 0.05 M  $\text{H}_2\text{SO}_4$  for 40 min under ultrasonic treatment of the slurry at  $20 \pm 2^\circ\text{C}$  and S : L = 1 : 10. After the conversion, the solid phase was separated by filtration, washed on the filter with three portions of distilled water at S : L = 1 : 5, dried in air for 8 h, and dissolved in a 1.0 M aqueous  $\text{Na}_2\text{CO}_3$  solution under ultrasonic treatment for 60 min at S : L = 1 : 10. The temperature in the course of dissolution increased to  $70^\circ\text{C}$ . The solution obtained was filtered to remove the undissolved precipitate and analyzed for the content of U(VI) and FP impurities. The decontamination factors were calculated as the ratio of the quotients from division of the impurity concentration by the U(VI) concentrations in the initial simulated SNF and in the carbonate solution obtained after its dissolution.

As expected, by dissolving both a pellet and a powder of simulated SNF we obtained solutions containing 80.5 and 74.9 g  $\text{L}^{-1}$  U(VI), respectively. Impurities of such elements as Mo(VI), Cs, Ca, Zr(IV), Ba, Sr, Al, and Cu completely and partially passed into the solution. The concentrations of REE, Cr, Mn, Pd, Sb, and Y did not exceed 0.1 mg  $\text{L}^{-1}$ . In accordance with the solubility, the decontamination factors from soluble

impurities (Mo, Cs, Ca, Cu) did not exceed 5, and those from poorly soluble impurities ranged from ~250 to 3600.

Further purification should be reached in the steps of extraction with QAC from the carbonate solutions obtained.

Thus, oxidative dissolution of uranium SNF in carbonate solutions in the presence of H<sub>2</sub>O<sub>2</sub> allows preparation of sufficiently concentrated solutions containing uranyl peroxide-carbonate complexes and concomitant readily or partially soluble FP impurities. In this step, the reached decontamination factors from poorly soluble impurities range from several hundreds to several thousands.

#### ACKNOWLEDGMENTS

The study was financially supported by the Russian Science Foundation (agreement no. 14-23-00188).

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*Translated by G. Sidorenko*