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Effect of Ionizing Radiation on the Extraction of Am(III) with *p-tert*-Butylthiacalix[4]arene from Alkaline Carbonate Solutions

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Abstract—The effect of γ-irradiation of *tert*-butylthiacalix[4]arene (TCA) solutions in *m*-nitrobenzotrifluoride (NBTF) and tetrachloroethylene (TCE) on the extraction of ^{241}Am from alkaline carbonate solutions was studied. TCA itself remains stable upon γ-irradiation of its solutions in NBTF to a dose of 200 kGy, but the diluent undergoes strong degradation. The radiation resistance of TCA in TCE is considerably lower: A dose of 70 kGy causes complete degradation of TCA. In the TCA–TCE–aqueous phase system, sulfate ions appear upon γ-irradiation as the final product of the extractant radiolysis. A large number of γ-radiolysis products of TCE and TCA were detected by HPLC and GCMS. The products of radiolysis of TCA in TCE, compared to the initial extractant, have lower molecular mass and higher polarity. The results show that chlorinated diluents are not promising diluents for thiacalixarene in extraction processing of alkaline high-level waste.

Keywords: extraction, americium-241, thiaxalix[4]arene, alkaline media, γ-radiolysis, radiolysis products

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One of the most important problems of modern applied radiochemistry is final liquidation of so-called “nuclear heritage.” Active efforts are made at nuclear objects for remediation of contaminated territories, decommissioning of large nuclear facilities, and final removal and disposal of the accumulated radioactive waste. One of the aspects of this problem is processing of high-level waste (HLW) accumulated in the course of implementation of the USSR Nuclear Project at the Mayak Production Association. This HLW has a volume of more than 18 000 m^3. It is in the form of multicomponent slurries containing sodium hydroxide, carbonate, nitrite, and nitrate in combination with such hazardous radionuclides as ^{137}Cs, ^{90}Sr, and transuranium nuclides [1–3].

The first step of processing of complex multicomponent HLW should involve recovery of hazardous radionuclides from the liquid phase of the heterogeneous medium by one or another procedure. This study was aimed at developing a process for extraction preconcentration and separation of ^{241,243}Am from multicomponent alkaline media using high-performance macrocyclic compounds. We have shown that Am(III) is efficiently extracted from alkaline carbonate solutions with substituted thiacalix[4]arenes. *p*-Bromothiacalix[4]arene at pH 12 showed the highest ability to extract Am (distribution ratio $D_{\mathrm{Am}} > 100$) and the highest selectivity [selectivity factor $\beta_{\max}(\mathrm{Am/Eu}) = 18$] [4].

The suggested promising extraction system for recovering radionuclides from alkaline carbonate media should be adapted to real conditions by performing trials with simulated HLW solutions under the action of ionizing radiation. First, it is necessary to evaluate the radiation resistance of the extractants and diluents used in the experiments, taking into account contradictory data published in the literature. For example, Kikuchi et al. [5] reported high radiation resistance of *p*-*tert*-butylthiacalix[4]arene: After irradiation to an absorbed dose of up to 1 MGy, the degree of degradation was as low as 1%. Furthermore, under definite conditions (pH 2–4) the ability of thiacalix[4]arene for coordination to Am(III) was enhanced, which was attributed to oxidation of sulfide bridges to sulfone groups in the course of irradiation. On the contrary, Špendlíková et al. [6] noted low radiation resistance of *p*-*tert*-butylthiacalix[4]arene in 1,2-dichloroethane: The degree of degradation was up to 99% at a dose of 10 kGy, whereas in cyclohexanone at a dose of 100 kGy under equal other conditions only 60% of the thiacalixarene decomposed.

In this study, we examined the effect of γ-irradiation on the extraction of Am(III) with solutions of *p*-*tert*-butylthiacalix[4]arene in *m*-nitrobenzotrifluoride and tetrachloroethylene from alkaline carbonate solutions.

EXPERIMENTAL

As diluents in solvent extraction and extractant irradiation experiments we used *m*-nitrobenzotrifluoride (NBTF) (Rhodia, France) and pure grade tetrachloroethylene (TCE) (Vekton, St. Petersburg, Russia), washed with a sodium carbonate solution and with water. Solutions of *p*-*tert*-butylthiacalix[4]arene in these diluents were prepared from the accurately weighed portion of the substance. Inorganic chemicals were of no worse than analytically pure grade (Vekton) and were used without additional purification. Solvents for HPLC and GPC (Acros Organics) were used without additional purification. ^{241}Am containing 99.9% main isotope was produced at the Khlopin Radium Institute.

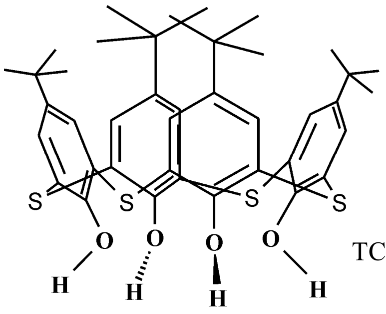
p-*tert*-Butylthiacalix[4]arene sample containing 95% main substance was synthesized and characterized by the known procedure [7]. *p*-*tert*-Butylphenol (99%) and sulfur (98%) used in the synthesis were produced by Acros Organics. All the chemicals and solvents were used in the synthesis without additional purification.

TCA/2 was synthesized as follows: A round-bottomed flask was charged with 5.00 g (33.33 mmol) of *p*-*tert*-butylphenol, 0.11 g (3.33 mmol) of sulfur, and 0.02 g (0.42 mmol) of NaOH. The mixture was stirred at 180°C for 6 h. After cooling to room temperature, 20 mL of hexane was added to the solidified mixture. The undissolved residue consisting of unchanged phenol and sulfur was filtered off, and hexane was removed from the filter on a rotary evaporator. The residue was dried in a vacuum for several hours, and TCA/2 was obtained as a beige powder in 51% yield. The product purity was checked by TLC (chloroform/hexane, 2 : 1, R_f 0.1). The spectroscopic data and melting point of the sample agreed with the published data [8].

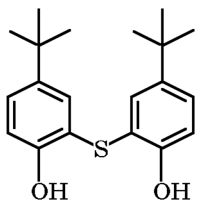
Calix[4]arene sulfone was prepared by the known procedure [9].

CAS/2 was synthesized by oxidation of TCA/2 dissolved in chloroform with a mixture of 30% H_2O_2 and glacial acetic acid on heating on a glycerol bath (T_{bath} = 100°C) for 2 h. CAS/2 was isolated from the reaction mixture in the form of a beige crystalline precipitate in 82% yield by extraction with chloroform. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.26 s (18H, *t*-Bu), 5.90 s (2H, OH), 6.95 d (^3J = 8 Hz, 2H, ArH), 7.50 d.d (^3J = 8 Hz, ^4J = 4 Hz, 2H, ArH), 7.60 d (^4J = 4 Hz, 2H, ArH). IR spectrum, ν, cm^{-1}: 3415 (OH), 2962 (CH), 1308, 1134 (SO_2). Mass spectrum (electrospray ionization): *m*/*z* 361.1 [M^- – 1]. Found, %: C 66.38, H 7.06, S 8.26. $C_{20}H_{26}O_4S$. Calculated, %: C 66.27, H 7.23, S 8.85. Melting point 210°C.

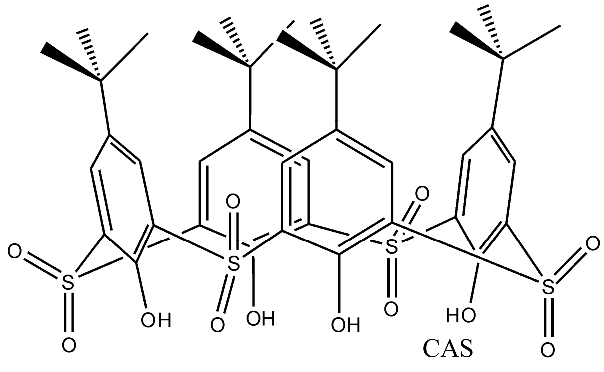
The formulas and designations of the extractants studied are given below.



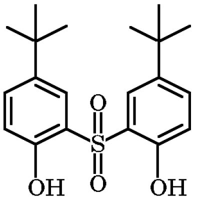
TCA



TCA/2



CAS



CAS/2

Experimental Procedures

^{1}H and ^{13}C NMR spectra. The ^{1}H and ^{13}C NMR spectra were recorded with a Bruker AVANCE 400 spectrometer. The mass spectra were taken on a Bruker amaZon X mass spectrometer with electrospray ionization (ESI) and recording of negatively charged ions. The IR spectra were measured with a Bruker Alpha FT–IR spectrometer from KBr pellets in the interval 500–4000 cm^{-1}. The melting points of the substances were determined on a Boetius small heating table with a PHMK 0.5 visual device. Elemental analysis was performed with a Perkin Elmer PE 2400 series 2 CHNS/O analyzer.

HPLC separation was performed with a Thermo Dionex UltiMate 3000 HPLC system. A Zorbax Eclipse Plus C18 RR column (particle size 3.5 μm, length 100 mm, inside diameter 4.6 mm) was used. Column temperature 35°C, flow rate 1.0 mL min^{-1}. The separation was performed in the isocratic mode in the acetonitrile/tetrahydrofuran (75 : 25) solvent system. The sample volume was 0.01 mL, and the concentration was 0.1 mg mL^{-1}.

Separation by gel permeation chromatography (GPC) was performed on an Agilent 1260 Infinity GPC device with an RI detector using two PLgel MiniMix-E columns (particle size 3 μm, length 250 mm, inside diameter 4.6 mm) with a PLgel MiniMix-E Guard protective column. THF for HPLC was used as a mobile phase at a flow rate of 0.3 mL min^{-1}. The sample concentration was ~1.0 mg mL^{-1}, and the sample volume, 0.02 mL. GPC analysis of the products was performed after 8-point calibration using polystyrene reference samples with narrow molecular-mass distribution in the molecular mass range from 580 to 10 100.

Studies by gas chromatography–mass spectrometry (GCMS) were performed with a Shimadzu GCMS-2010 Ultra device under the following conditions: column thermostat temperature 60°C, injector temperature 250°C, injection with flow splitting, pressure 34.8 kPa, total flow rate 106.0 mL min^{-1}, flow rate in the column 1.98 mL min^{-1}, linear velocity 51.3 cm s^{-1}, purging rate 5.0 mL min^{-1}, flow split ratio 50.0; thermostat temperature program: 2 min at 60°C, heating to 180°C at a rate of 10°C min^{-1}, 10 min at 180°C, and heating to 200°C at a rate of 10°C min^{-1}; MS parameters: ion source temperature 210°C, interface temperature 220°C, solvent subtraction time 2.50 min, detector amplification 0.96 kV, and scanned mass range 44–800.

Determination of the concentration. The concentration of sulfate ions in the aqueous phase after the irradiation was determined on a Staier ion chromatograph equipped with a TS10 column thermostat, a conductometric detector, and Mul'tiKhrom program–apparatus complex. The detection limit of sulfate ions was 0.2 mg L^{-1} (0.002 mM).

pH of solutions was determined with an HI-8314 digital pH meter (Hanna Instruments) using an I-1330B microelectrode allowing the measurements to be performed in the extraction micro test tubes. The pH meter was calibrated using standard buffer solutions with pH 7.01 and 10.01.

Extraction of Am(III). The distribution ratios of radionuclides were determined as follows: A 1.5-mL centrifugal polypropylene micro test tube was charged with 0.6 mL of an aqueous phase of preset composition. The aqueous phases with a preset pH value were prepared by mixing 1 M $NaHCO_3$ and NaOH solutions in appropriate ratio; constant ionic strength of the solutions was thus ensured. The equilibrium pH value was determined directly in the test tube. After that, a radioactive tracer was added, the components were mixed, and the mixture was allowed to stand for 15 min. Then, 0.6 mL of the organic phase was added, and the phases were stirred at 21–23°C for 15 min. Preliminary experiments showed that this time was sufficient to attain the equilibrium in the system. The phase separation was performed by centrifugation at 3000 rpm for 10 min. We took for measurements 0.4-mL samples of the organic and aqueous phases. The Am content was determined radiometrically from the γ-radiation of ^{241}Am. The distribution ratios were calculated by the formula $D_{\mathrm{Am}} = A_{\mathrm{org}}/A_{\mathrm{aq}}$, where A is the activity concentration of the radionuclide in the corresponding phase. The extraction was studied with tracer amounts of ^{241}Am. Radiometric measurements were performed with a DeskTop InSpector scintillation γ-ray spectrometer (Canberra) based on a well-type 51 × 51 mm NaI detector. The measurement time was chosen so as

Table 1. List of irradiated samples and compositions of the aqueous and organic phases

Sample no.	Absorbed dose, kGy	Aqueous phase[a]	Organic phase
1	70	–	1 mM TCA in TCE
2		H_2O	
3		1	
4		2	
5		3	
6	200	–	Pure TCE
7		–	1 mM TCA in TCE
8		H_2O	
9		1	
10		2	
11		3	
12		1	Pure TCE
13		2	
14		3	

[a] (1) 0.6 M NaOH + 0.4 M $NaHCO_3$, (2) 0.45 M NaOH + 0.3 M $NaHCO_3$ + 0.25 M $NaNO_3$, and (3) 0.45 M NaOH + 0.3 M $NaHCO_3$ + 0.25 M $NaNO_2$.

to ensure no more than 10% uncertainty of radiometric measurements.

Extractant irradiation. Irradiation was performed in glass test tubes with stoppers equipped with gas release tubes. The volume of each phase in the test tube was 5 mL. Prior to irradiation, the phases were thoroughly mixed. Irradiation was performed to absorbed doses of 70 and 200 kGy. The irradiated samples are listed in Table 1. The extractants were irradiated on an Issledovatel' γ-ray installation (Mayak Production Association) using a ^{60}Co source with the total activity of 6.1×10^{14} Bq and maximal γ-radiation dose in the center of the working chamber of 14 kGy h^{-1}.

After the irradiation completion, the phases were mixed. After the emulsion separation, we took from each test tube 4.5 mL of the aqueous phase to determine the concentration of sulfate ions and 4.5 mL of the organic phase for further studies.

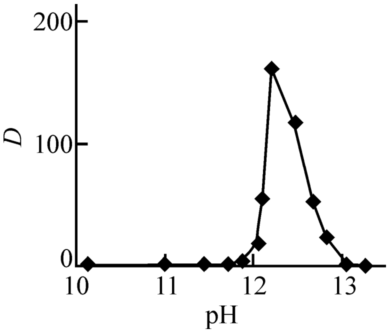


Fig. 1. Extraction of Am(III) from alkaline carbonate solutions with a 2×10^{-4} M solution of TCA in NBTF.

RESULTS AND DISCUSSION

One of factors limiting the use of calixarenes in extraction processes is their low solubility in organic solvents used in solvent extraction. However, low solubility of *tert*-butylthiacalix[4]arene in NBTF (~10^{-4} M) does not prevent the use of this reagent for efficient extraction of Am(III) from an alkaline carbonate medium (Fig. 1). As can be seen, the Am(III) extraction maximum is at pH in the interval from 12 to 13, i.e., TCA in NBTF can be used for extracting radionuclides from alkaline HLW.

Preliminary experiments on γ-irradiation of a solution of TCA in NBTF demonstrated high radiation resistance of the thiacalixarene, in agreement with the data of [5]. In the two-phase system consisting of 2×10^{-4} M TCA in NBTF and 1 M NaOH + 1 M Na_2CO_3 in the aqueous phase, at an absorbed dose of 200 kGy, the TCA concentration (according to HPLC data) did not change noticeably. γ-Irradiation of NBTF in contact with an alkaline aqueous phase leads to accumulation of black radiolysis products in the organic phase and complicates determination of the Am(III) distribution ratios in extraction from alkaline carbonate solutions. Therefore, further experiments on TCA radiolysis were performed using TCE as diluent. This choice is due to high solubility of the reagent and convenience of studying the mixtures by spectroscopic and chromatographic methods. In addition, TCE is structurally similar to hexachlorobutadiene, a commercial diluent that was used at the Mayak Production Association for HLW processing.

In the next step of the study, a 1 mM solution of TCA in TCE and two-phase systems containing in the aqueous phase sodium hydroxide, carbonate, nitrate, and nitrite (components of alkaline HLW) were subjected to γ-irradiation (Table 1). The γ-irradiation dose in the experiment was chosen so as to reach the absorbed dose of 70 kGy, corresponding to the radiation load on the solvent in industrial processing of HLW before withdrawal from the cycle. After irradiation to a dose of 200 kGy, both the TCA–TCE organic phase and aqueous phases of all the compositions acquire yellow color. The color of the extractant samples obtained without contact with the aqueous phase and

Table 2. ^{241}Am distribution ratios in extraction with a 1 mM solution of TCA in TCE from alkaline carbonate solution (1 M of the sum NaOH + $NaHCO_3$, pH 13.44)

Sample no.	Aqueous phase, M	Irradiation dose, kGy		
		0	70	200
	Unwashed organic phase			
1	Without contact with aqueous phase	1.63	1.75	28.46
2	H_2O	–	2.67	2.5
3	0.6 NaOH + 0.4 $NaHCO_3$	–	1.65	1.96
4	0.45 NaOH + 0.3 $NaHCO_3$ + 0.25 $NaNO_2$	–	1.01	0.86
5	0.45 NaOH + 0.3 $NaHCO_3$ + 0.25 $NaNO_3$	–	1.1	1.72
	Organic phase washed with two portions of 0.5 M Na_2CO_3			
6	Without contact with aqueous phase	2.2	1.94	1.27
7	H_2O	–	1.08	0.61
8	0.6 NaOH + 0.4 $NaHCO_3$	–	1.45	2.56
9	0.45 NaOH + 0.3 $NaHCO_3$ + 0.25 $NaNO_2$	–	0.67	1.3
10	0.45 NaOH + 0.3 $NaHCO_3$ + 0.25 $NaNO_3$	–	1.18	1.78

Table 3. Content of SO_4^{2-} in the equilibrium aqueous phase after γ-irradiation of a 1 mM solution of TCA in TCE

Aqueous phase, M	Concentration of SO_4^{2-} in equilibrium aqueous phase after irradiation, mM	
	70 kGy	200 kGy
0.6 NaOH + 0.4 $NaHCO_3$	0.035	0.11
0.5 NaOH + 0.3 $NaHCO_3$ + 0.25 $NaNO_2$	0.48	0.37
0.45 NaOH + 0.3 $NaHCO_3$ + 0.25 $NaNO_3$	0.83	0.75

in contact with pure water was the most intense.

For the irradiated and nonirradiated samples of the organic phase, we determined D_{Am} under similar conditions. Some of the experiments were performed with unwashed samples of the irradiated organic phases, and other experiments, with the organic phases washed after irradiation with two portions of a 0.5 M Na_2CO_3 solution at the phase volume ratio of 1 : 1. Data on the extraction of Am(III) from alkaline carbonate solutions with nonirradiated and irradiated samples of TCA solutions in TCE are given in Table 2. As can be seen, in many cases D_{Am} increased after irradiation of the organic phase. For example, after irradiation of a TCA solution without contact with the aqueous phase to a dose of 200 kGy (sample 1 in Table 2), D_{Am} increased by a factor of 20 compared to the unirradiated sample of the same composition. On the contrary, washing of the irradiated organic phase with a Na_2CO_3 solution in most cases decreased D_{Am}. Presumably, TCA degradation products extract Am(III) more efficiently than TCA itself does, but are partially washed out from the organic phase in the course of sodium carbonate washing, which is confirmed by the data noted in [5].

Chemical analysis of some samples taken from the aqueous phases after the TCA irradiation revealed the presence of sulfate ion, the final product of TCA radiolysis (Table 3). As can be seen, the maximal yield of sulfate ions in the aqueous phase does not exceed 20% of the theoretically possible yield. Hence, complete degradation of TCA does not occur even at an irradiation dose exceeding by a factor of 3 the dose reached in the course of industrial extraction processing of HLW.

Because the formation of calixarene sulfone (CAS) and its degradation products (CAS/2) in the course of the TCA radiolysis via oxidation of sulfide bridges could be expected, we have studied the extraction of Am(III) with these compounds (Fig. 2).

As can be seen, CAS and CAS/2 extract Am from alkaline solutions with the distribution ratios that are lower than those in extraction with TCA by a factor of several thousands. Thus, in alkaline solutions oxidation of TCA to CAS cannot cause an increase in D_{Am}, in contrast to weakly acidic solutions [5].

Another possible pathway of TCA radiolysis is the TCA macroring opening with the formation of linear products. To confirm this assumption, we studied the Am extraction from alkaline solutions with a solution

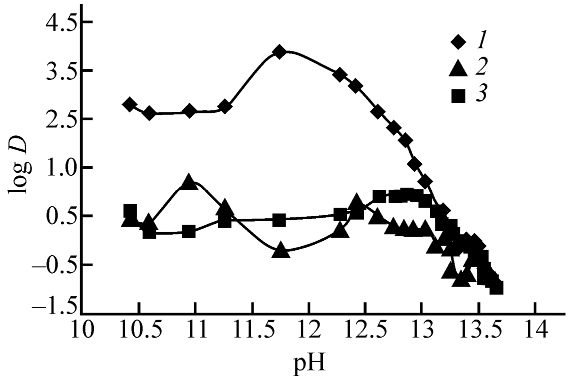


Fig. 2. Extraction of ^{241}Am with 1 mM solutions of (*1*) TCA and (*2*) CAS and (*3*) with a 2 mM solution of CAS/2 in TCE from alkaline carbonate solutions in relation to pH of the aqueous phase.

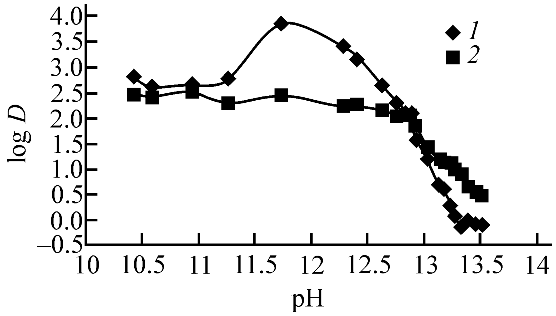


Fig. 3. Extraction of Am(III) from alkaline carbonate solutions with (*1*) 0.001 M solution of TCA and (*2*) 0.002 M solution of TCA/2 in TCE.

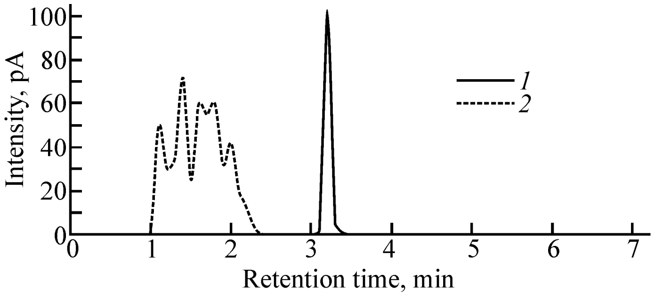


Fig. 4. Chromatograms (reversed-phase HPLC) of a solution of TCA in TCE (*1*) before irradiation and (*2*) after irradiation to a dose of 200 kGy.

of TCA and its nonmacrocyclic analog, TCA/2, which is a “half” of the thiacalixarene molecule (Fig. 3). As can be seen, TCA/2 exhibits very high extraction ability, quantitatively extracting Am from the aqueous phase at its pH up to 13. The Am distribution ratios at high pH values in the extraction with thiacalixarene and its nonmacrocyclic analog, TCA/2, differ insignificantly (Fig. 3).

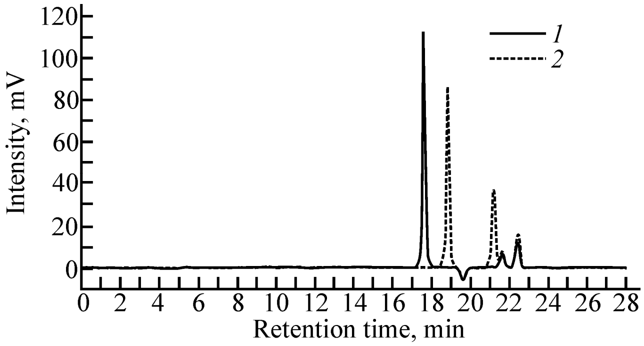


Fig. 5. Chromatograms (GPC) of a solution of TCA in TCE (*1*) before irradiation and (*2*) after irradiation to a dose of 200 kGy.

In the final step of the study, we studied the TCA and TCE radiolysis products by HPLC and GCMS. We studied irradiated solutions of TCA in TCE by HPLC using a reversed-phase column. As we found, TCA is absent in these samples, and the retention times of the radiolysis products are shorter than that of the initial TCA, suggesting their higher polarity. The chromatograms are shown in Fig. 4.

To evaluate the molecular masses of products formed by radiolysis of solutions of TCA in TCE, the irradiated solutions were analyzed by GPC. We have found that the irradiated TCA solutions do not contain substances with the relative molecular mass higher than 300 amu (Fig. 5).

A GCMS analysis of irradiated solutions of TCA in TCE revealed the presence of products formed by radiolysis of TCE itself and by subsequent transformation of the primary radiolysis products: penta- and hexachloroethane, perchloro-1,3-butadiene, octachloropropane, and hexachloropropene, which could be formed by transformations of highly reactive radical intermediates ($Cl^{\bullet}$, $CCl_2{=}CCl^{\bullet}$) generated by radiolysis of C–Cl bonds. In the case of thiacalixarene, the formation of various chlorinated phenols and thiobisphenols is observed.

The γ-irradiation-induced transformations of lower unsaturated chlorocarbons presumably consist in detachment of chlorine atoms and their addition to molecules of the substance being irradiated. The radicals formed upon detachment of chlorine atoms probably recombine or undergo addition to the initial substance with the subsequent detachment of chlorine atoms [6, 10–12]. However, the processes occurring upon TCE irradiation are insufficiently understood yet.

Thus, we have demonstrated the stability of TCA upon γ-irradiation of its solution in nitrobenzotrifluoride in the presence of an alkaline aqueous phase to an absorbed dose of up to 200 kGy. In the process, nitrobenzotrifluoride undergoes significant radiolysis. After irradiation of a TCA solution in TCE, D_{Am} in the extraction of Am(III) increases, although TCA is not detected in the organic phase. Probably, TCA degradation products extract Am(III) more efficiently than TCA itself does.

A study of possible TCA degradation products has shown that calix sulfone CAS and its linear analog CAS/2 do not extract Am, whereas the nonmacrocyclic analog of TCA is similar in the extraction ability to TCA. γ-Irradiation of the system consisting of TCA in TCE and alkaline aqueous phase to a dose of 200 kGy leads to the formation of sulfate ion in amounts corresponding to complete degradation of approximately 20% of TCA.

HPLC and GCMS analysis shows that the radiolysis products of TCA solution in TCE have lower molecular mass and higher polarity compared to the initial compound. In the irradiated organic phase, we detected the products formed by radiolysis of TCE itself and by the subsequent chemical transformations of the primary radiolysis products: penta- and hexachloroethane, perchloro-1,3-butadiene, octachloropropane, and hexachloropropene.

The diluents checked in this study are unsuitable for extraction processing of alkaline HLW: NBTF undergoes strong radiation degradation, and TCE radiolysis products cause the TCA degradation.

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