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Yttrium-90 recovery from carbonate media with binary extractants based on hydroxyaromatic compounds and methyltrioctylammonium carbonate

Ahmed H. A. Harb[1] · **Igor V. Balantsev**[3] · **Maria D. Karavan**[2,3] · **Igor V. Smirnov**[2,3]

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Abstract
A new method of yttrium recovery with a mixture of hydroxyaromatic compound (8-hydroxyquinoline, 2,3-dihydroxynaphthalene) and methyltrioctylammonium carbonate separation from strontium in carbonate media has been proposed. Yttrium extraction from carbonate media has been studied. The structures of yttrium solvates with ligands formed during extraction have been proposed. The suggested method for yttrium recovery from soil by sample calcination followed by carbonate leaching and extraction with the specified mixture allows the efficient separation of yttrium-strontium pair and yttrium recovery.

Keywords Solvent extraction · Carbonate media · Methyltrioctylammonium carbonate · Yttrium-90 · 8-hydroxyquinoline · 2,3-dihydroxynaphthalene

Introduction

Strontium-90 (^{90}Sr) is one of the biologically significant radionuclides. It is an osteotropic element, capable of accumulating mainly in the skeleton, entering the body of animals and humans with food [1]. When ^{90}Sr enters the soil, it quickly becomes involved in the physicochemical processes occurring there [2].

Strontium plays a leading role in the pollution of natural water reservoirs. Monitoring its content in surface and underground waters is especially important in places where nuclear power cycle enterprises are located, and where radioactive waste is stored and disposed of [3]. There are some difficulties associated with the presence of high concentrations of macrocomponents. This has caused interest in searching for methods of more selective radionuclide concentration. For example, in paper [4] the physicochemical and sorption properties of the carbonate containing zirconium hydroxide based T-3 K sorbent were studied, which showed the prospects of its use for concentrating ^{90}Sr from natural waters of various compositions. The authors developed a method for ^{90}Sr determining in natural waters with the ^{90}Sr concentration on the T-3 K sorbent, and tested the method by ^{90}Sr monitoring in natural reservoirs and in control and observation wells of a radioactive waste storage facility.

The ^{90}Sr activity determination in soils is usually carried out by oxalate and express-extraction radiochemical methods, based on determining the activity of the daughter ^{90}Y by their joint extraction from the soil. The oxalate method, which takes at least two weeks to implement, requires the radioactive equilibrium attainment between yttrium and strontium. The express extraction method, which takes one to two days to implement, does not require the radioactive equilibrium attainment and uses a solution of HDEHP (bis(2-ethylhexyl)phosphoric acid) in heptane with additional purification of the extracted ^{90}Y with Aliquat 336 solution in toluene [5]. The use of HDEHP for the Sr/Y separation has also been developed in modern research, for example, in a variant of adsorption separation of Sr/Y on silica-based bis(2-ethylhexyl) hydrogen phosphate adsorbent [6].

✉ Maria D. Karavan
mkaravan@khlopin.ru

[1] Hot Laboratories Center, Radioisotopes Production and Radiation Sources Division, Nuclear Chemistry Department, Egyptian Atomic Energy Authority, P.O. Box 13759, Cairo, Egypt

[2] Institute of Chemistry, Saint-Petersburg State University, Universitetskaya emb., 7–9, Saint-Petersburg, Russia 199034

[3] Khlopin Radium Institute, 2nd Murinsky Pr., 28, Saint-Petersburg, Russia 194021

In radioanalytical methods of analysis, crown ethers on various carriers, are actively used as selective extractants of strontium [7–12]. For example, SR Resin (TRISKEM Int.) with crown ether (4,4′(5′)-di-t-butylcyclohexyl-18-crown-6) dissolved in octanol is used as an extractant [7]. According to the papers [8, 9], the best efficiency of strontium separation from the SR Resin is achieved when using 7 mol L^{-1} and 0.05 mol L^{-1} HNO_3 as eluents. Then, a new porous crown-based resin (DtBuCH18C6@CG-71) with efficient ^{90}Sr capture from highly acidic wastewater was synthesized by introducing 4′,4″(5″)-di-*tert*-butyldicyclohexano-18-crown-6 onto CG-71 resin using vacuum impregnation. This resin is regarded as promising for ^{90}Sr disposal in nuclear wastewater [10]. Another recent study described dibenzo-18-cown-6 functionalized chloromethyl-polystyrene (CMPS-DB18C6) resin with an excellent affinity toward Sr^{2+} ion adsorption from aqueous solution [11].

Being a daughter product of the decay of ^{90}Sr, ^{90}Y can be used for the ^{90}Sr determination in natural samples [12–14]. Among the 20 known radioactive isotopes of yttrium, ^{90}Y is the most important one. It is extremely in demand for cancer therapy due to its nuclear physical properties: a short half-life (64.05 days) and almost pure β^--decay with maximum energy $E_{max}(\beta^-) = 2.28$ MeV [15]. To obtain carrier free ^{90}Y, it is chemically isolated from an equilibrium system with the parent radionuclide ^{90}Sr, one of the main fission products of uranium. Carrier free ^{90}Y can also be produced from uranium fission products, but it is contaminated with ^{91}Y [16].

The standard method of chemical isolation of ^{90}Y is solvent extraction. The extraction of ^{90}Y and its separation from ^{90}Sr have been studied for acidic media using dioctylphosphoric acid [17], beta-diketones [18], di(2-ethylhexyl)phosphoric acid (D2EHPA) [19–21] and crown ethers [22, 23]. The methods for yttrium separation from strontium and its recovery described above are based on the preliminary technique of acid leaching, which involves the use of toxic and aggressive reagents, and are also time-consuming due to the necessity of the radioactive equilibrium attainment for the pair ^{90}Y/^{90}Sr. In contrast to this, we proposed a procedure for yttrium and strontium recovery from carbonate media [24], taking into account that, for strontium, the possibility of its recovery from carbonate media using polyfunctional *p*-alkylcalix[8]arenes has already been explored [25].

The main goal of the present work is to study the fundamental possibility of using an alternative carbonate method for yttrium isolation, which consists in carbonate leaching of yttrium with its further separation from ^{90}Sr by extraction using a binary mixture based on hydroxyaromatic compounds and methyltrioctylammonium carbonate (MTOAC).

Experimental

Materials and techniques

Two hydroxyaromatic compounds were proposed as potential extractants of yttrium in carbonate media: 2,3DHN (2,3-dihydroxynaphthalene) (Schuhardt, Germany) and 8HQ (8-hydroxyquinoline) (Vekton, Russia) (Fig. 1). All organic diluents: 2-nitrotoluene (Lancaster, England), butyl acetate, and toluene (Ekos-1, Russia) were of analytical grade and were used without any further purification. $SrCO_3$ (Reakhim, Russia) was of analytical grade; yttrium carbonate stock solution was prepared by dissolving yttrium oxide in analytical grade potassium carbonate solution (0.05 mol L^{-1} K_2CO_3). MTOAC was synthesized at the Department of Technology of Rare Elements and Nanomaterials Based on them of the Mendeleev Russian University of Chemical Technology of Russia by the original method and provided by Dr. S.I. Stepanov. Radioactive isotopes ^{90}Y and ^{90}Sr were received from the Khlopin Radium Institute (Russia).

The extraction experiments were conducted as follows: the aqueous and organic phases (1 mL each) were placed into a 4 mL centrifugal polypropylene tube, and then the two-phase systems contacted at 21 ± 1 °C for 10 min. Then the two-phase systems were centrifuged at 3000 rpm for 5 min to enhance phase separation. The distribution ratios (D), extraction percent (%E), and separation factor (SF) were calculated through: $D = \frac{C_{org}}{C_{aq}} = \frac{C_i - C_{aq}}{C_{aq}}$, $\%E = \frac{C_{org}}{C_i}$, $SF = \frac{D_1}{D_2}$, where C_{org} and C_{aq} are the metal concentrations after extraction in the organic and aqueous phases, C_i is the initial metal concentration, D_1 and D_2 are the distribution ratios of metal 1 and 2, respectively.

The pH of the aqueous phase was measured by an Expert pH-meter (± 0.5 °C, pX (pH) ± 0.005, mV ± 0.2).

Yttrium concentration in aqueous phases was determined by several methods:

- by Arsenazo III standard spectrophotometric method ($l = 1$ cm, $\lambda_{max} = 650$ nm) using UV-2600 SHIMADZU UV–VIS spectrophotometer,

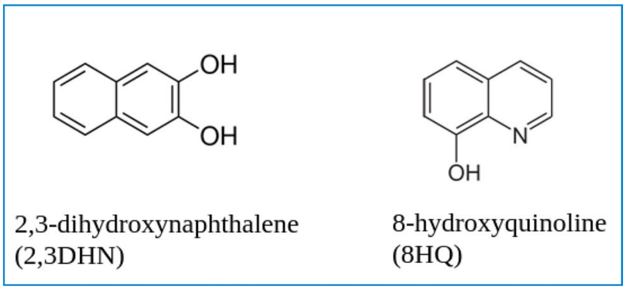

Fig. 1 Structural formulae of potential yttrium extractants

- by ICP-OES, ICPE-9000 SHIMADZU emission spectrometer.

Concentrations of strontium and other metals were determined by ICP-OES, ICPE-9000 SHIMADZU emission spectrometer.

Structural studies of yttrium complexes were conducted by XRD (X-ray diffractometer Shimadzu labX XRD 6000) and by attenuated total reflectance infrared spectroscopy ATR-FTIR (FT-801 Simex analytical equipment).

Radioactive isotopes ^{90}*Sr and* ^{90}*Y* were determined by liquid scintillation counting and Cherenkov counting standard methods using low level liquid scintillation spectrometer Quantulus 1220 (PerkinElmer, 4π- geometry, uncertainty less than 10%) with a valid calibration certificate. Standard radioactive solution of ^{90}Sr and ^{90}Y (in equilibrium) with a valid calibration certificate was used as a comparison sample. EasyView software was used for imaging beta spectra.

The ^{90}*Y counting in analyzing samples* was additionally measured on a Teflon target with an A85 radiometer (plastic scintillator, 2π geometry, count efficiency ~ 25%, uncertainty ~ 5% at stated exposition time). All counting samples were prepared by applying an aliquot of the ^{90}Y containing aqueous solution to a Teflon target, evaporating the aqueous phase under an IR lamp, and then analyzing with a radiometer. Comparison of the counting rate of the samples and their activity in Bq was made by measuring a counting sample of a standard ^{90}Y solution of known activity.

Leaching experiments were conducted with a soil sample (500 g). The soil was homogenized, sieved, and spiked with a solution of ^{90}Y/^{90}Sr pair in radioactive equilibrium (the specific activity of the soil was about 1 Bq g^{-1}). The soil sample with the spike was dried in an oven at 80 °C for several days; after which a 2 g sample was taken. Solutions of stable yttrium and strontium nitrates in a molar ratio of 10:1 were added to this sample, components were thoroughly mixed and dried in an oven. Then, Na_2CO_3 and K_2CO_3 were mixed with the soil sample (10 g of each carbonate) and the mixture was annealed 1 h at 300 °C, then 4 h at 550 °C. The cooled mixture was entered into a polypropylene test-tube with leaching agent, and after 2 h was taken for yttrium extraction. Yttrium was extracted from treated soil sample with a mixture of 0.01 mol L^{-1} 2,3DHN and 0.015 mol L^{-1} MTOAC in toluene. Yttrium was stripped with 0.05 mol L^{-1} HNO_3 solution (O:A = 4:1). Yttrium concentration and ^{90}Y specific activity in aqueous phase were analyzed.

Results and discussion

Yttrium extraction. According to the previous experiments, we chose methyltrioctylammonium carbonate as the special modificator. Among several examined ligands [18, 20], only 2,3DHN and 8HQ demonstrated appreciable synergism with MTOAC (Table 1).

Table 1 shows that the addition of MTOAC made it possible to avoid the formation of the third phase for both hydroxyaromatic ligands in toluene and only for 8HQ in 2-nitrotoluene. Therefore, for further tests, we chose butyl acetate and toluene as the main organic diluents.

We used the method of constant molar ratio to gain more insight into the synergistic effect of 2,3DHN/MTOAC and 8HQ/MTOAC on yttrium extraction. The total molar concentration of the mixed extractant (MTOAC and ligands) was kept constant at 0.005 mol L^{-1} (Fig. 2). D_Y in the mixed extractant changed with the mole fraction of the components.

The highest yttrium extraction for the system 2,3DHN/MTOAC was obtained at the 1:1.5 molar ratio, whereas the

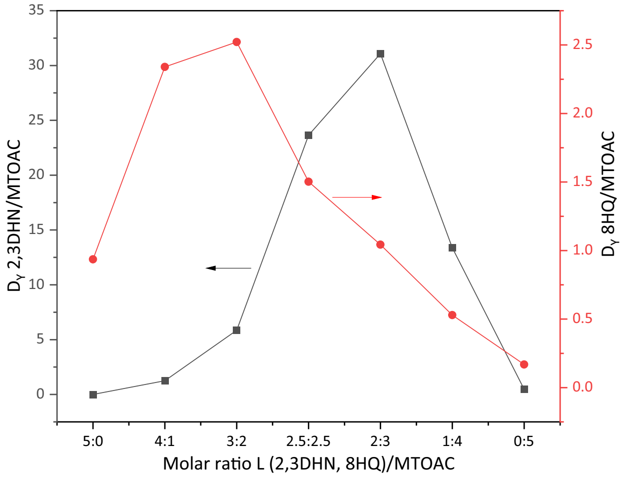

Fig. 2 Dependence of D_Y on molar ratio of components in a mixed extractant (in toluene). C_Y = 0.001 mol L^{-1}. pH = 12

Table 1 D_Y values for 0.01 mol L^{-1} 2,3DHN, 8HQ with and without 0.015 mol L^{-1} MTOAC (pH = 12)

Ligand (L)	D_Y					
	L + MTOAC			L		
	BuAc	Toluene	2-nitrotoluene	BuAc	Toluene	2-nitrotoluene
8HQ	87	85	3rd phase	66	3rd phase	73
2,3DHN	83	85	3rd phase	3.2	3rd phase	3rd phase

highest one for the system 8HQ/MTOAC was observed at the 1.5:1 molar ratio.

MTOAC cation forms hydrophobic salts with phenolate anion and decreases the extractant wash-out to aqueous phase. The stoichiometry of yttrium extraction with 2,3DHN or 8HQ and MTOAC could be expressed as follows:

$$[Y(CO_3)_n]^m + xL + z(R_4N)_2CO_3[YL_X(CO_3)_{n-x}]^z \cdot z(R_4N)_2 + (z + (n-x))CO_3^{2-}$$

where $m=(3-2n)$, $z=3-2(x+(n-x))$ and L = 2,3DHN or 8HQ.

The fact that the optimal ratio of components in the extraction mixture is a 1.5 fold excess of MTOAC over 2,3DHN suggests the formation of a solvate with the composition $[Y(2,3DHN)_2(MTOAC)_3]$. Our preliminary studies using MS–ESI(-) confirmed this assumption: in addition to the 2,3DHN monoanion, the spectra contain the signal of the $[Y(2,3DHN)_2]^-$ monoanion, as well as those of anionic complexes with carbonate, hydroxyl groups and water molecules [26].

The influence of pH was studied on yttrium(III) extraction in the range 10–13.5 (Fig. 3).

2,3DHN/MTOAC and 8HQ/MTOAC demonstrated high D_Y values in both organic diluents. With increasing pH, the D_Y dramatically increased and reached values over 50 at pH 11.5. It is noteworthy that the $\log D_Y$ values for 2,3DHN achieved a plateau already at pH 10–10.5.

The extraction isotherm was constructed by plotting the yttrium equilibrium concentration in the organic phase versus that in the aqueous phase (Fig. 4).

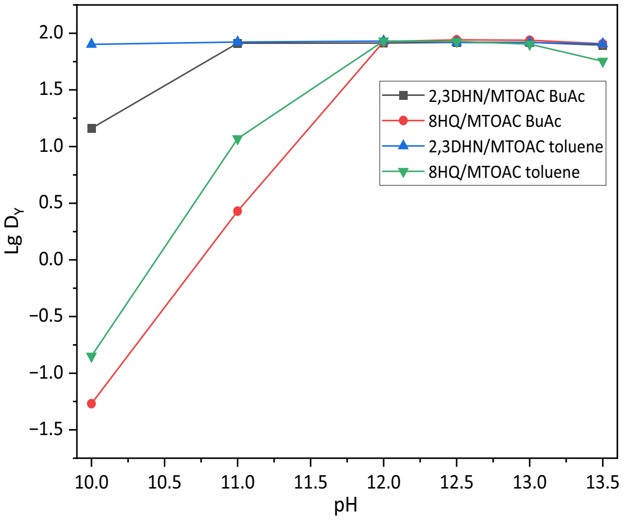

Fig. 3 Dependence of D_Y on pH for yttrium extraction from carbonate media. $C_Y=0.001$ mol L^{-1}, $C_L=0.01$ mol L^{-1}, (L-2,3DHN or 8HQ), $C_{MTOAC}=0.015$ mol L^{-1}

Yttrium extraction, using toluene and BuAc as diluents, with a synergistic mixture of 0.01 mol L^{-1} 2,3DHN and 0.015 mol L^{-1} MTOAC, was studied. At maximum loading, the molar ratio of synergistic mixture and metal was about 3 in toluene and 4 in BuAc.

Yttrium-strontium separation. The Y(III)/Sr(II) separation factor (SF) was calculated for the following extraction system: aqueous carbonate solution of 4×10^{-6} mol L^{-1} Y(III) and 1×10^{-6} mol L^{-1} Sr(II), and organic phase with 1×10^{-3} mol L^{-1} MTOAC and 1.5×10^{-3} mol L^{-1} 2,3DHN in toluene or BuAc (Fig. 5).

Y/Sr separation factor $SF_{Y/Sr}$ in extraction from aqueous carbonate media with 2,3DHN in toluene demonstrated

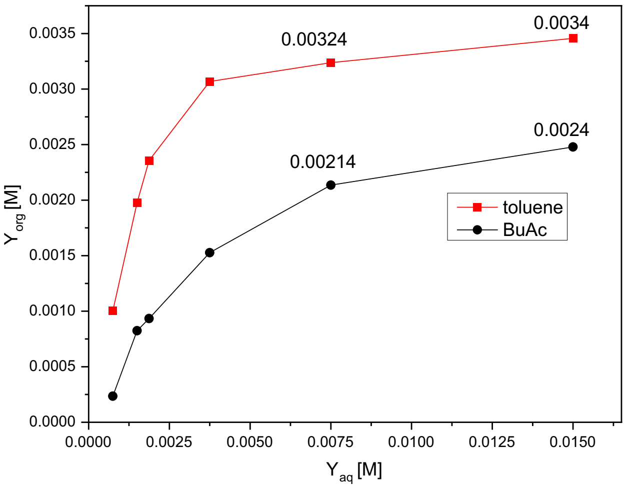

Fig. 4 Yttrium extraction isotherm, constructed for the mixture of 0.01 mol·L^{-1} 2,3DHN and 0.015 mol L^{-1} MTOAC in toluene and BuAc (pH = 12)

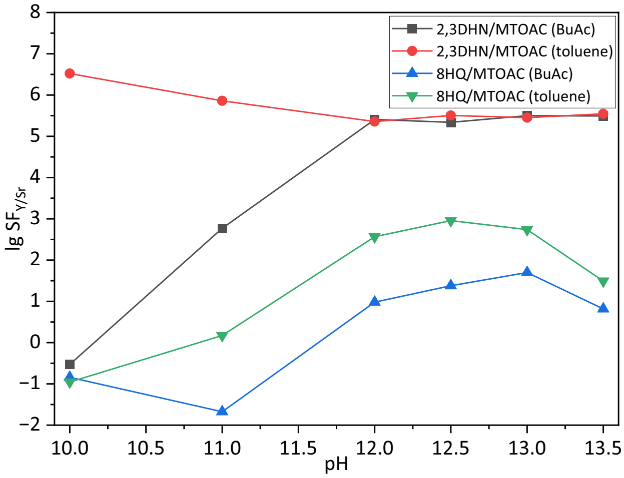

Fig. 5 Yttrium–strontium separation with extraction system of 1×10^{-3} mol L^{-1} 2,3DHN or 8HQ and 1.5×10^{-3} mol L^{-1} MTOAC from the aqueous carbonate media (4×10^{-6} mol L^{-1} Y(III) and 1×10^{-6} mol L^{-1} Sr(II))

steady values in all pH ranges ($\log SF_{Y/Sr} = 5.5$), while in BuAc it increases with pH up to pH = 12 (maximum $\log SF_{Y/Sr} = 5.5$). The values of $\lg SF_{Y/Sr}$ for 8HQ reached 2.96 and 1.67 in toluene and BuAc, respectively, with pronounced maximum at pH interval 12–13.

Structure of extractable complexes. We correlated XRD, ATR-FTIR and extraction data to figure out the Y-2,3DHN configuration.

To obtain Y-2,3DHN crystalline substance, we conducted an extraction experiment in methyl acetate with a simple extraction system without MTOAC (BuAc has low volatility, MTOAC retards crystal growth); after washing and re-crystallization in EtOH, the sample was analyzed by XRD. Its structure appeared as a sandwich-like structure, where yttrium was located between the layers of 2,3DHN (Fig. 6).

To further confirmation of the extracted complex structure, we used ATR-FTIR, which can provide insight into how different ligands interact with metals in the extraction process. To study the metal-ligands interaction in the extraction process, binary mixtures of extractant (2,3DHN, 8HQ) and MTOAC were prepared. A small amount of ligands was dissolved in MTOAC, and the freshly prepared ligands were diluted in toluene to extract yttrium from carbonate media. After yttrium extraction, toluene was evaporated, and a small amount of the substance was analyzed by ATR-IR (Fig. 7).

As one can see, MTOAC has a band at 1381 cm^{-1} due to asymmetric stretching vibrations of O–C–O in CO_3^{2-} [27, 28]. The band attributed to C–O dramatically decreased after interaction with ligands. A new band attributed to C–H and C–C stretching vibration of 2,3DHN was detected at 1265 cm^{-1}. C–N band of MTOAC appeared at 1460 cm^{-1}, the sharp absorption bands at 2928 and 2855 cm^{-1} were assigned to the C–H stretching vibration, whereas the ring bending is observed at 850 cm^{-1}. The ~ 730 and 700 cm^{-1} bands were assigned to stretching modes of Y-ligands bond. In both systems, the symmetric and asymmetric stretching vibration of H_2O band at 3400 cm^{-1} decreased. This band was accounted for the washout of incorporated H_2O in MTOAC after interaction with ligands and disappeared after extraction as a result of water transfer to aqueous phase. The intensities and positions of absorption bands are affected when Y^{3+} forms a complex with ligands. It is clear that, in comparison with the ligand's spectra, all Y^{3+} complexes exhibit an increase in intensity of bands between 1200 and 1500 cm^{-1}. We believe that this effect is caused by electron density transfer from the ligand to the metal ion [29–31].

Leaching experiments on laboratory and real samples

In order to examine our method on real samples, we carried out the experiment on yttrium leaching and extraction from soil.

Our previous experiments showed that yttrium can be easily stripped from the organic phase with HNO_3, HCl and H_2SO_4 solutions. The best results were obtained for 0.05 mol L^{-1} HNO_3 (even one contact was enough for quantitative yttrium stripping). Moreover, after four cycles of extraction and stripping, the extraction efficiency of the mixture based on 2,3DHN was almost constant, and the extractant did not need regeneration, which is not the case for the system, based on 8HQ [20].

Based on the results of our recent experiment on leaching yttrium from sand and several soil samples with aqueous solutions, we selected the optimal soil treatment conditions.

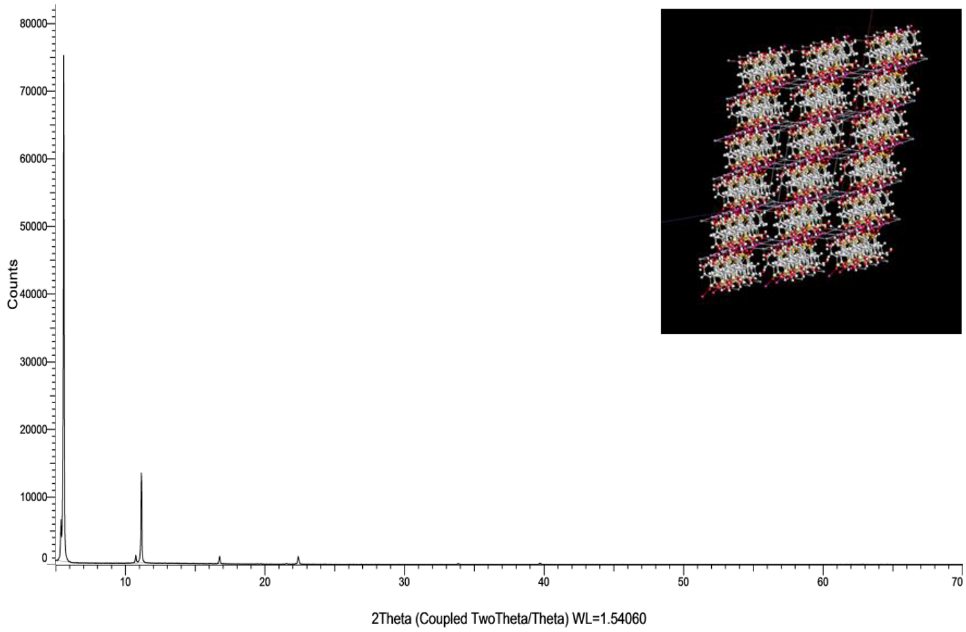

Fig. 6 XRD patterns of Y-2,3DHN

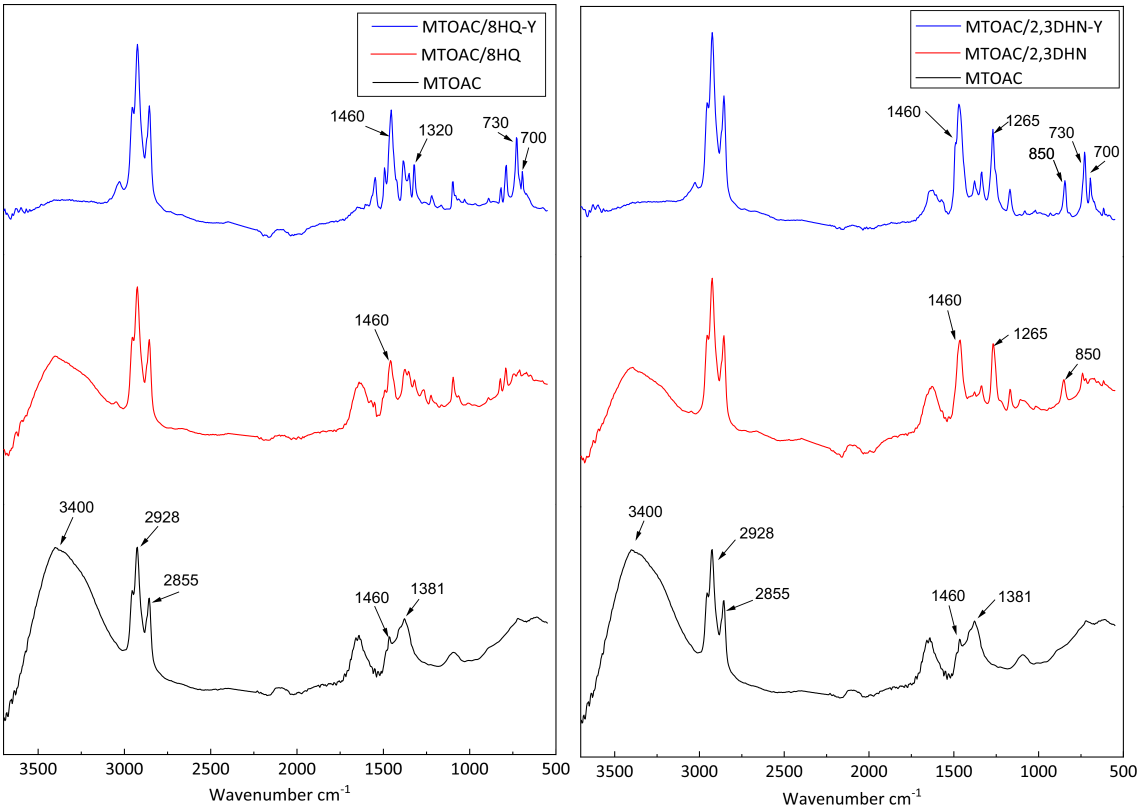

Fig. 7 The ATR-IR spectra of ligands and their yttrium complexes

In our recent experiments, several compounds (hydrogen peroxide and citric acid) were used to increase yttrium leachability. Citric acid forms mononuclear, binuclear, or polynuclear, as well as bi-, tri- and multidentate complexes depending on the type of metal ion being chelated. In addition, citric acid is a relatively safe and non-toxic substance. Adding hydrogen peroxide can help to oxidize organic compounds. Experiments have shown that H_2O_2 does not affect yttrium leaching, while citric acid enhances yttrium leaching. Another option for increasing yttrium leaching was to replace the leaching agent with an alkaline solution. One of the possible transformations during the calcination process is the formation of metal oxides, so we also used 1 mol L^{-1} $(NH_4)_2CO_3$ as an alkaline leaching agent to prevent the formation of metal hydroxides [20]. All these data were used to select new calcination conditions followed by leaching and extraction processes. The main results are presented in Tables 2 and 3.

Table 2 shows that citric acid enhanced yttrium leaching. The best convergence of yttrium separation results was found for the system with potassium carbonate and citric acid (68% by spectrophotometry and 65–67% radiometrically). 71% yttrium recovery, obtained by spectrophotometric determination with Arsenazo III, seems to be overestimated because of possible side reactions with other trivalent cations leached from the soil.

Since the initial idea of sintering the mixture with carbonates during the calcination process did not lead to success, the calcination was carried out without adding a mixture of carbonates. The results of yttrium extraction show that the process of leaching and subsequent extraction of yttrium from the solution with the proposed extraction mixture is almost identical in efficiency to that one with preliminary addition of carbonates at the calcination stage (Table 3).

A comparison of the yttrium extraction data, presented in Tables 2 and 3 shows that the yttrium can be recovered from soil without the stage of alkaline sintering, which simplifies the analysis (Fig. 8).

The mixture 1 mol L^{-1} K_2CO_3/0.3 mol L^{-1} citric acid as a leaching agent demonstrates the smallest errors (2.5 and 5.5%) in ^{90}Sr determination by ^{90}Y, both in the case of preliminary sintering with carbonates and in the case of simple soil sample calcination before leaching.

Conclusions

The study of the extraction properties of a mixture based on a hydroxyaromatic compound and methyltrioctylammonium carbonate revealed the following main features of the process:

Table 2 The effect of citric acid on yttrium recovery from soil after alkaline carbonate leaching (soil sintering with carbonates during the calcination process followed by the alkaline leaching)

Leaching agent	Spectrophotometric determination		Radiometric determination (plastic scintillator)		Radiometric determination (LSC)	
	C_Y, mg L^{-1}	% of recovery	A_Y, Bq g^{-1}	% of recovery	A_Y, Bq g^{-1}	% of recovery
	Initial 19.5 ± 0.2		Initial 1.00 ± 0.02		Initial 1.00 ± 0.02	
1 mol L^{-1} NH_4HCO_3/ 0.3 mol L^{-1} citric acid	13.8 + 0.3	71	0.53 ± 0.02	53	0.56 ± 0.03	56
1 mol L^{-1} $NaHCO_3$/ 0.3 mol L^{-1} citric acid	13.0 ± 0.2	67	0.51 ± 0.03	51	0.57 ± 0.04	57
1 mol L^{-1} K_2CO_3/ 0.3 mol L^{-1} citric acid	13.3 + 0.2	68	0.65 + 0.04	65	0.67 ± 0.04	67

Table 3 The effect of citric acid on yttrium recovery from soil after alkaline carbonate leaching with different carbonates (simple soil calcination without any carbonates followed by the alkaline leaching)

Leaching agent	Spectrophotometric determination		Radiometric determination (plastic scintillator)		Radiometric determination (LSC)	
	C_Y, mg L^{-1}	% of recovery	A_Y, Bq g^{-1}	% of recovery	A_Y, Bq g^{-1}	% of recovery
	Initial 19.5 ± 0.2		Initial 1.00 ± 0.02		Initial 1.00 ± 0.02	
1 mol L^{-1} NH_4HCO_3/ 0.3 mol L^{-1} citric acid	13.2 ± 0.3	68	0.54 ± 0.02	54	0.55 ± 0.03	55
1 mol·L^{-1} $NaHCO_3$/ 0.3 mol L^{-1} citric acid	13.6 ± 0.3	70	0.51 ± 0.37	51	0.56 ± 0.01	56
1 mol L^{-1} K_2CO_3/ 0.3 mol L^{-1} citric acid	12.7 ± 0.3	65	0.61 ± 0.18	61	0.62 ± 0.02	62

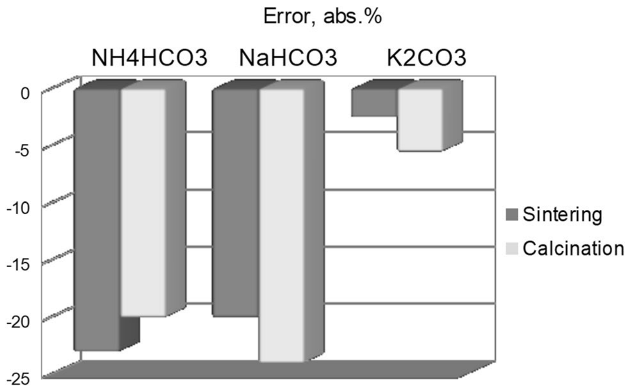

Fig. 8 Determination of ^{90}Sr by its daughter ^{90}Y in soil after calcination (sintering), alkaline carbonate leaching, and selective extraction using 2,3DHN/MTOAC in toluene

- the working pH range for the effective Y/Sr separation and subsequent yttrium recovery is the interval 11.5–13.5;
- yttrium is most effectively extracted with a mixture of 2,3-dihydroxynaphthalene and MTOAC in toluene or butyl acetate with a component ratio of 2,3-DHN:MTOAC = 1:1.5;
- the most efficient Y/Sr separation occurs when yttrium is extracted with a mixture of 2,3-DHN and MTOAC in toluene or butyl acetate (logSF = 5.5 in both cases);

A possible structure of yttrium solvates with the 2,3DHN ligand has been proposed, and it is a sandwich-like structure with yttrium between the ligand layer.

A study of the possibility of yttrium recovery from a soil showed that at least 60% of the yttrium contained in a sample can be extracted by successive sintering, carbonate leaching, and extraction. In this case, there is good agreement between the analysis results obtained with stable (spectrophotometry) and radioactive yttrium. Incomplete yttrium recovery from the soil may be due to shortcomings in the chosen sample preparation and is the subject of further study. The proposed method can be useful not only for Y/Sr separation for preparative purposes, but also for strontium analysis in the natural samples as one of the main markers of radioactive contamination.

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Data availability The authors confirm that the data supporting the findings of this study are available within the article and its supplementary materials.

Declarations

Conflict of interest All authors declare that they have no conflicts of interest.

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