


Post-recoil thermal annealing study of ^{177}Lu , ^{169}Yb , ^{175}Yb , ^{166}Ho and ^{153}Sm in different organometallic compounds

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Abstract. Post-recoil thermal annealing study at different temperatures for different intervals of time of the recoiled isotopes ^{169}Yb , ^{175}Yb , ^{177}Lu , ^{166}Ho , and ^{153}Sm resulting from the nuclear reactions $^{168}\text{Yb}(n,\gamma)^{169}\text{Yb}$, $^{174}\text{Yb}(n,\gamma)^{175}\text{Yb}$ and $^{176}\text{Yb}(n,\gamma)^{177}\text{Yb} \rightarrow ^{177}\text{Lu}$, $^{165}\text{Ho}(n,\gamma)^{166}\text{Ho}$, $^{152}\text{Sm}(n,\gamma)^{153}\text{Sm}$, respectively in different organometallic compounds $\text{Yb}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$, $\text{Ho}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$, $(\text{C}_5\text{H}_5)_3\text{Ho}$, and $(\text{C}_5\text{H}_5)_3\text{Sm}$ were carried out. The thermal annealing behaviors of ^{177}Lu , ^{169}Yb , ^{175}Yb , ^{166}Ho , and ^{153}Sm were found to be different in the compounds under investigation. The differences in the patterns of the obtained isothermal curves were discussed. Depending on the post-recoil thermal annealing manner the reactions taking place during thermal annealing were suggested.

Key words: thermal annealing • lutetium • ytterbium • holmium • samarium

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Introduction

Apart from the most recent physical applications in the development of various superconducting materials, many compounds of the rare earth metals have been investigated with regard to their biochemical and pharmaceutical properties [16]. A number of rare earth elements have physiological properties which may be of interest in the diagnosis or treatment of certain cancers, and several β -emitting isotopes of the lanthanide series, such as ^{177}Lu , ^{169}Yb , ^{175}Yb , ^{166}Ho and ^{153}Sm have been evaluated for a variety of nuclear medical applications [15].

As a result of historical revision of post-recoil thermal annealing [1, 2, 4–14], it was found that although thermal annealing phenomena have been studied in a variety of compounds, no attempt had been made in the case of organometallic lanthanides [15, 16]. As the most important objectives of the study of annealing reactions are to understand reaction mechanisms and to identify the nature and the environment of species produced in the nuclear reactions, the experiments conducted in this work aimed to analyse and explain the thermal annealing in order to understand the recoil mechanism in organometallic lanthanides as well as the mechanism of parent reformation in these compounds.

Experimental and procedures

Materials

Two organometallic metallocenes triscyclopentadienyl holmium and triscyclopentadienyl samarium and two organometallic betadiketones *tris*(2,2,6,6-tetramethyl-

-3,5-heptanedionato)Ho(III) and *tris*(2,2,6,6-tetramethyl-3,5-heptanedionato)Yb(III) were obtained from Alfa. All chemical solvents used were of analytical reagents (AR) grade. Solvents were dried using MgSO_4 before using to prevent any scavenger action as a result of the presence of moisture in chromatography solvents due to the fact that the compounds under investigation are moisture sensitive. Whatman Chr. 1 and Whatman Chr. 4 chromatographic paper were used for paper chromatography separation.

Irradiation

A weighed amount of about 5 mg was irradiated in a low power tank in a pool research reactor MNSR (miniature neutron source reactor) for 1–8 h at a thermal neutron flux of $10^{12} \text{ n}\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$ and at a temperature of approximately 50°C . The inner polyethylene capsule was encapsulated into an outer polyethylene capsule. Both of them were closed by thermal welding. The tightness of the inner capsules was examined by putting them in a hot water bath. $\text{Ho}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$ and $\text{Yb}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$ were encapsulated in the laboratory atmosphere, while $(\text{C}_5\text{H}_5)_3\text{Ho}$ and $(\text{C}_5\text{H}_5)_3\text{Sm}$, which are moisture and oxygen sensitive, were encapsulated in an inert dry atmosphere of nitrogen (99.999%) using a glove box designed for this purpose.

Thermal annealing and separation procedures

Post irradiation thermal annealing at different temperatures 50, 100, 150 and 200°C for different intervals of time of 1, 3, and 5 h was performed in an electronically controlled oven. The organic and inorganic forms were separated from the irradiated targets by paper chromatography using Whatman Chr. 1 and Whatman Chr. 4 dimensions of $1.5 \times 10 \text{ cm}$. THF was used as a solvent and as a developer for both of $(\text{C}_5\text{H}_5)_3\text{Ho}$ and $(\text{C}_5\text{H}_5)_3\text{Sm}$, while for $\text{Yb}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$ toluene was used as a solvent and acetone as a developer, for $\text{Ho}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$ acetone was used as a solvent and toluene as a developer. The latest compound was also separated by extraction using toluene and water with the addition of holmium chloride HoCl_3 as a carrier, where it was dissolved in HCl (0.05 M).

Counting procedure

Radioactivities were measured by high resolution γ -ray spectrometry using high purity germanium (HPGe) detectors with a different relative efficiency of 40 and 60%. To avoid errors resulting from variations in counting geometry, all measurements were carried out using the same position and orientation of the chromatographic paper on the detector as well as the distance from it. With respect to sublimation all samples were counted using equal volumes in reproducibly positioned identical vials. Finally, all counting data were corrected for the dead time.

Results and discussion

Thermal annealing at 50°C

Thermal annealing at 50°C was carried out for two different compounds $\text{Yb}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$, and $(\text{C}_5\text{H}_5)_3\text{Sm}$. The thermal annealing at 50°C for $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$, and $(\text{C}_5\text{H}_5)_3\text{Sm}$ were shown in Fig. 1, where the values, in all cases, represent averages of several independent experiments. As shown in Fig. 1, both of isothermal curves were hardly changed within 5 h of heating. These two different compounds had obviously different initial retention values before the thermal annealing, taking into consideration that the retention is defined as: $R = [A_{\text{org}}/(A_{\text{org}} + A_{\text{inorg}})]$, where A_{org} is the activity of the organic separated species, and A_{inorg} is the activity of inorganic separated species. The very small change in retention value during 5 h of thermal annealing at 50°C could be explained for $(\text{C}_5\text{H}_5)_3\text{Sm}$ compound assuming that no recombination reaction could happen at this relatively low temperature. Although there is a theoretical possibility of recombination depending on the structure and chemical bonds, for $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ compound, it seemed that recombination reactions reached the saturation at a temperature of 50°C during the neutron irradiation, where 50°C represents the temperature in the irradiation positions [3]. The big difference in the retention values between the two compounds could be explained by two factors. Firstly, the differences which related to the two different metals samarium and ytterbium like recoil energy, cation radii, electron configuration. Secondly,

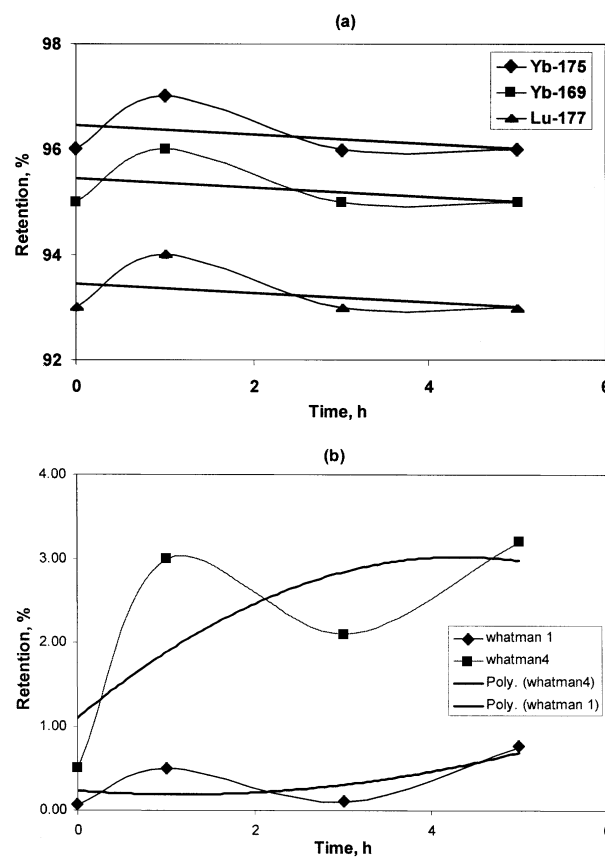


Fig. 1. Thermal annealing at 50°C for $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ (a) and $(\text{C}_5\text{H}_5)_3\text{Sm}$ (b) compounds.

the differences which are related to the organic part of the compound, like the chemical pounds, as well as the spatial structure (i.e., in complex compounds such as $\text{Yb}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$, the recoil energy has a good chance of being dissipated through the molecule before the recoil atom can leave its immediate neighborhood (i.e. failure of bond rupture may be particularly significant in such cases of complex molecules, and the probability of being the direction of recoil atom to be directed toward the center of mass of the molecule, it is likely that a large amount of the energy could be absorbed within the molecule, at least long enough for the atoms to remain in essentially their normal configurations while the energy is transferred to neighboring molecules). The presence of sigma pounds in $\text{Yb}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$ means that there is a probability that at least some part of the molecule remained intact after the nuclear event, or at least the ligand remained intact. With respect to π -bonded compounds like $(\text{C}_5\text{H}_5)_3\text{Sm}$, the distinguishing feature is that a fragmented ligand can no longer give rise to the same type of compounds, indeed, to a stable compound at all. While it is possible that atoms of C and O may rapidly re-form a CO molecule, which is then able to react further, the fragments of cyclopentadiene cannot be expected to arrange themselves so as to reform the original or any other π -bonding ligand. The molecule is thus destroyed unless it can be reformed by an exchange reaction, or replaced by another molecule in which one or more ligands is σ -bonded [13].

In $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ the difference between the resulting radioactive isotopes ^{175}Yb , ^{169}Yb , and ^{177}Lu was clear with respect to the initial retention values. For this compound, using either Whatman Chr. 1 or Whatman Chr. 4 gave the same results, but it was found that the development using Whatman Chr. 4 was faster. While for $(\text{C}_5\text{H}_5)_3\text{Sm}$ using different types of chromatographic papers gave different results. The retention values when using Whatman Chr. 4 were little bit more comparing with Whatman Chr. 1. Furthermore, the retention value differences between Whatman Chr. 1 and Whatman Chr. 4 became larger by increasing the period of heating. The difference in the retention values using Whatman Chr. 1 and Whatman Chr. 4 might be explained as follow; different kinds of recoiled species, which are affected by the type of chromatographic paper, were produced as a result of hot reactions in $(\text{C}_5\text{H}_5)_3\text{Sm}$, while these recoiled species did not appear in $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$. This reasonably could be happened because of the earlier mentioned differences between $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ and $(\text{C}_5\text{H}_5)_3\text{Sm}$. In addition, the species which were developed by Whatman Chr. 4 are more sensitive for the duration of the thermal annealing than those which are developed by Whatman Chr. 1 which made the difference between retention values obtained by two types of the chromatographic papers to become larger with increasing duration of heating.

Thermal annealing at 100°C

Thermal annealing at 100°C was carried out for all the compounds under investigation. The thermal annealing curves for $\text{Ho}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$, which was

separated by extraction and paper chromatography, showed a different behavior for different methods of separation. This difference might be due to the solid-state interaction between the water-extractable fraction and the toluene-soluble fraction, where it was found that heating of the irradiated ferrocene at 110°C causes a sharp decrease in the water-extractable activity (from 55 to 15% in 2–3 h). This 40% difference did not appear as ferrocene and was assumed to form some stable molecular species other than ferrocene [13].

In addition to the fact that the parent organometallic compound was dissociated under effect of the acidic pH, where HCl (0.05 M) was to be added for complete carrier HoCl_3 dissolution. This suggestion was proved by simulate the extraction process using cold $\text{Ho}(\text{OCC}(\text{CH}_3)_3\text{CHCOC}(\text{CH}_3)_3)_3$ (without irradiation) via the UV spectra of the separated water and toluene. In the spectra of the separated toluene new wave-lengths were observed which did not belong to the compound under investigation. These new wave-lengths most likely belong to the new compounds as a result of dissociation.

In our case within 2–3 h there was an increase in annealing curve of about 15%, after the retention value decreased. Within 5–8 h, the retention value was stable and almost equal to the initial retention value (retention at time zero), in this stage thermal decomposition of the compounds could not be excluded. On the other hand, the difference between the couple of isothermal curves using extraction might be due to the cooling time effect, where one of the experiments was carried out after 24 h after at the end of irradiation, while the second experiment was carried out after 47 h after at the end of irradiation.

The difference in the manner between annealing curves, which were followed by extraction and by paper chromatography, for the same compound could also be explained by the changes of extent and even the nature of initial recoil species during extraction. As the annealing curves of the same compound, using two different methods of separation, were different, the first conclusion which could be made is that the final thermal annealing curves were affected by the method of separation. On the other hand, with respect to the thermal annealing followed by paper chromatography of $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Ho}$ there was a slow, but linear increase of retention of about 15% with increasing time of annealing. A comparison between thermal annealing followed by both extraction and paper chromatography for $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Ho}$ was shown in Fig. 2. The manner of slow but linear increase of retention with an increase in the duration of heating could be explained by recrystallization according to the hot zone model. This model suggests that the hot zone during cooling contains disorder of various types of broken bonds, free radicals, interstitial atoms, and vacancies. Annealing process regarded as the one of recrystallization, where the disordered region would be a second phase of the same crystal, thermodynamically unstable with respect to the first phase, which is the normal crystal. Since the two phases are in contact, the normal matrix serves as the nucleus for recrystallization [5].

Isothermal curve belongs to $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ compound in Fig. 3 followed the classical pattern, i.e., an

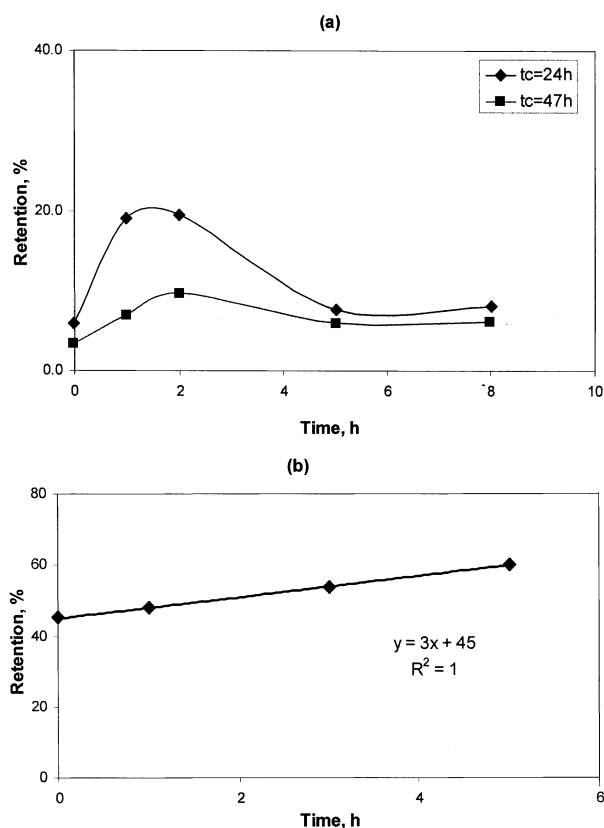


Fig. 2. Thermal annealing at 100°C for $(C_{11}O_2H_{19})_3Ho$ followed by extraction (a) and paper chromatography (b).

initial sharp rise followed by a temperature dependent pseudoplateau. The annealing plateau was reached in about 1 h. The difference between the initial retention values of ^{169}Yb , ^{175}Yb as well as ^{177}Lu became smaller by increasing of the annealing time. This phenomenon became clearer when the compound was annealed at 150°C. It could be explained using Harbottle's idea, namely, if a strong isotope effect is shown, then very little subsequent chemical influence can be felt and the observed species must be formed by prethermal processes. The supposition here is that isotopic differences come only from the differences in the nuclear deexcitation pattern (total energy, γ -ray cascades, angular correlation, conversion coefficients, etc.) and that subsequent thermal reactions will have no tendency to preserve memory of the nuclear event. The classi-

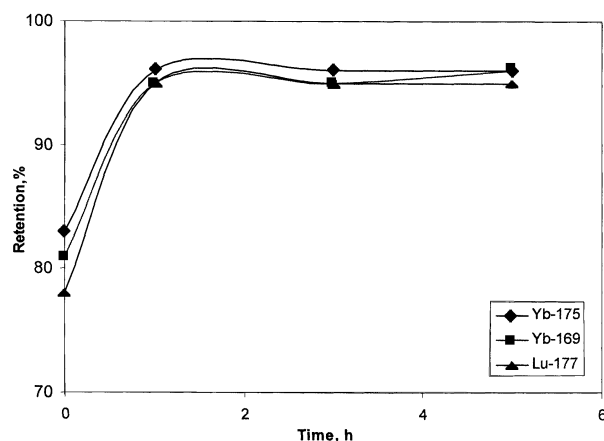


Fig. 3. Thermal annealing at 100°C for $(C_{11}O_2H_{19})_3Yb$.

cal isothermal curve could be explained as a simple recombination of correlated pairs, where the annealing process consists of the recombination of the recoil atom with a collision fragment or with one of its originally bonded partners [5]. Although the nature of the curves belonging to $(C_{11}O_2H_{19})_3Yb$ was similar to that in earlier studies [2, 4, 10–12] the saturation time for other compounds like chloropyridine cobaloxime, $KMnO_4$, $K_2Cr_2O_7$, Fe-Phthalocyanine, $La(MnO_4)_3$ respectively are different. Such differences were considered as a consequence of the variation of recoil energy, chemical pounds and spatial structure.

The difference between the thermal annealing at 100°C for the two similar compounds $(C_{11}O_2H_{19})_3Ho$ and $(C_{11}O_2H_{19})_3Yb$ followed by paper chromatography could be explained by the difference of electron configuration, as well as the difference of recoil energy of each of the different recoiled isotopes which, as a result, affect the kind of resulting recoiled species, which in its turn affect the kind of reactions which can happen during thermal annealing, and, as a result, the final curve will be different. In addition to this, the difference of Ho and Yb radii and their nature of the cations could be the reasons for giving different retention values, too.

Almost a similar trend of the isothermal annealing curves has been shown by $(C_5H_5)_3Ho$ and $(C_5H_5)_3Sm$. With respect to $(C_5H_5)_3Sm$, the retention difference for 5 h of heating was about 2% using Whatman Chr. 4, but had a neglected difference value during the same period of annealing time using Whatman Chr. 1. While for $(C_5H_5)_3Ho$, using both Whatman Chr. 1 and Whatman Chr. 4, the resulting curves had a neglected difference during 5 h heating. It is clear that both of them were affected by the type of chromatographic paper.

Suggesting that the most probable recoiled species were $C_{10}H_{10}Ho^+$ and $C_{10}H_{10}Sm^+$, in addition to the positive charge, as a result, of internal conversion effects annealing curves could be explained by the recombination of correlated pairs with electron transfer as the rate-determining step model. In this model the partial or complete neutralization of the charge may be necessary before the recoil fragment can recombine with other fragments during annealing, and this electron transfer process may be the rate-determining step. In such systems the fragments which re-form the parent species may be trapped sufficiently close to one another for recombination which occurs when the recoil fragment has gained the required number of electrons from its surroundings. Nevertheless, alternatively the parent species might be re-formed from the reduced recoil fragment during the subsequent chemical reactions.

Also in comparison to the already known annealing curves patterns, it is possible that it happened a kind of recombination of correlated pairs after a random-walk process. In this model the probability of jumping of the interstitials, which are located outside the deformed region, in any direction is the same. These interstitials wander according to a random-walk process, those that wander back into the deformed region are subsequently captured by their original partners, and the rest wander off through the crystal [5].

The difference in the annealing curve manner between $(C_5H_5)_3Ho$ and $(C_5H_5)_3Sm$ could be attributed, as in the previous case, to the difference in electron

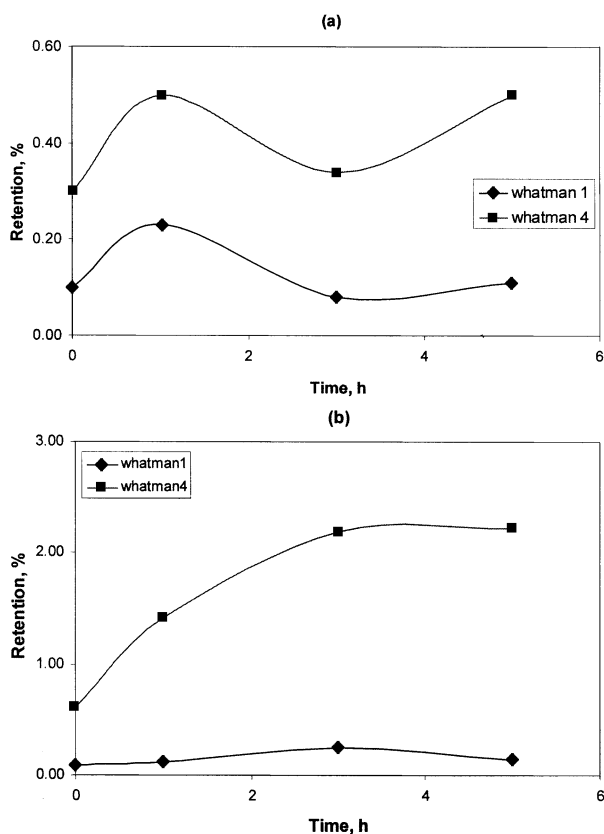


Fig. 4. Thermal annealing at 100°C for $(\text{C}_5\text{H}_5)_3\text{Ho}$ (a) and $(\text{C}_5\text{H}_5)_3\text{Sm}$ (b) compounds.

configuration, cation radii, and recoil energy between Ho and Sm. The thermal annealing at 100°C and a comparison between $(\text{C}_5\text{H}_5)_3\text{Ho}$ and $(\text{C}_5\text{H}_5)_3\text{Sm}$ is shown in Fig. 4.

Thermal annealing at 150 and 200°C

As a result of $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Ho}$ thermal annealing at 150°C for 1 h, the retention decreased to 22%, and for 3 h of annealing the retention decreased to 7%, for 5 h of annealing the compound decomposed. For $(\text{C}_5\text{H}_5)_3\text{Ho}$, the compound decomposed under heating within only 1 h. With respect to $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$, the obtained thermal annealing curve showed an increase of the retention value during the first hour, after which the retention value decreased under thermal decomposition. $(\text{C}_5\text{H}_5)_3\text{Sm}$, the most relatively thermal stable compound with respect to the rest of compounds under investigation showed a similar curve to that one obtained during annealing at 100°C with a slight increase in the retention values. The annealing of $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ and $(\text{C}_5\text{H}_5)_3\text{Sm}$ for 1 h at 200°C led to thermal decomposition of each of them. The thermal annealing curves of $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ and $(\text{C}_5\text{H}_5)_3\text{Sm}$ at 150°C are shown in Fig. 5.

Chromatographic paper type effect

Similar manner of isothermal annealing curves was observed with respect to $(\text{C}_5\text{H}_5)_3\text{Sm}$ in both cases using both types of chromatographic paper Whatman Chr. 1, and Whatman Chr. 4, however little bit

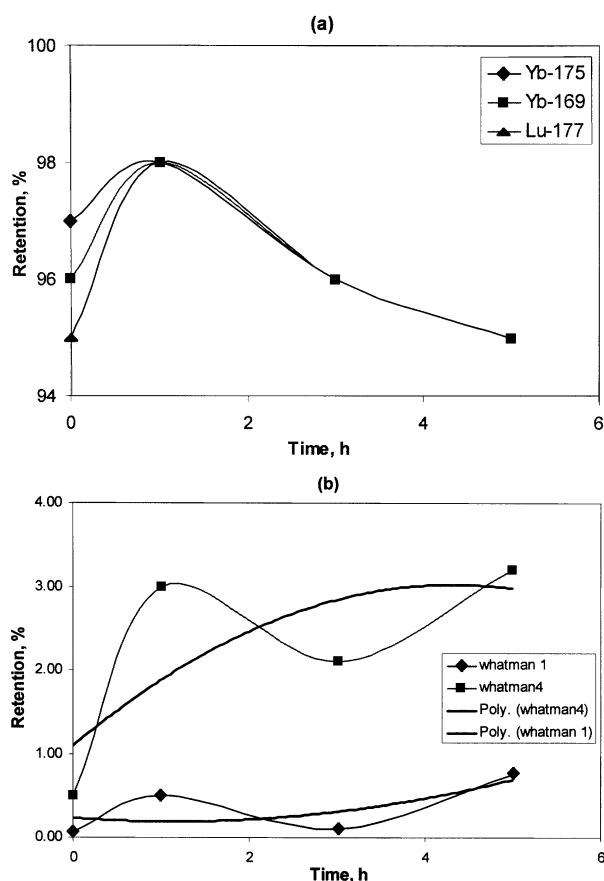


Fig. 5. Thermal annealing at 150°C for $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ (a) and $(\text{C}_5\text{H}_5)_3\text{Sm}$ (b) compounds.

higher retention values obtained when using Whatman Chr. 4. This could be explained by the presence of different kinds of species which could be developed by Whatman Chr. 4 and could not be developed by Whatman Chr. 1. These kinds of species resulted during nuclear recoil of each of $(\text{C}_5\text{H}_5)_3\text{Ho}$, and $(\text{C}_5\text{H}_5)_3\text{Sm}$ and did not result during nuclear recoil of each of $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Ho}$ and $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$, which seems to be logical as a result from former discussions. The effect of chromatographic paper type for $(\text{C}_5\text{H}_5)_3\text{Sm}$ at different temperatures 50, 100, and 150°C is presented in Fig. 6.

From the obtained isothermal annealing curves, it could be concluded that both $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Ho}$ and $(\text{C}_{11}\text{O}_2\text{H}_{19})_3\text{Yb}$ involve thermal reactions, $(\text{C}_5\text{H}_5)_3\text{Sm}$ involves, in part, thermal reactions, $(\text{C}_5\text{H}_5)_3\text{Ho}$ did not involve thermal reactions. It is because of the fact that any compound whose yield is influenced by thermal treatment involves at least, in part, thermal reactions. These thermal reactions can be of two types: those which led to the addition of further ligands, and those which do not because of simple failure of reaction as a result of the diffusion of the ligand from the reaction site, or because of stabilization of an intermediate stage by scavenger action, or there may be, at increased temperature, thermal dissociation of an intermediate product [13].

When only one type of ligand is present, it is clear that the same ligand type may react both prethermally and thermally. As $(\text{C}_5\text{H}_5)_3\text{Ho}$ was very small affected by thermal annealing, this suggests, in according with Harbottle's axiom, that most of $(\text{C}_5\text{H}_5)_3\text{Ho}$ were prethermally formed, where it is plausible to consider a

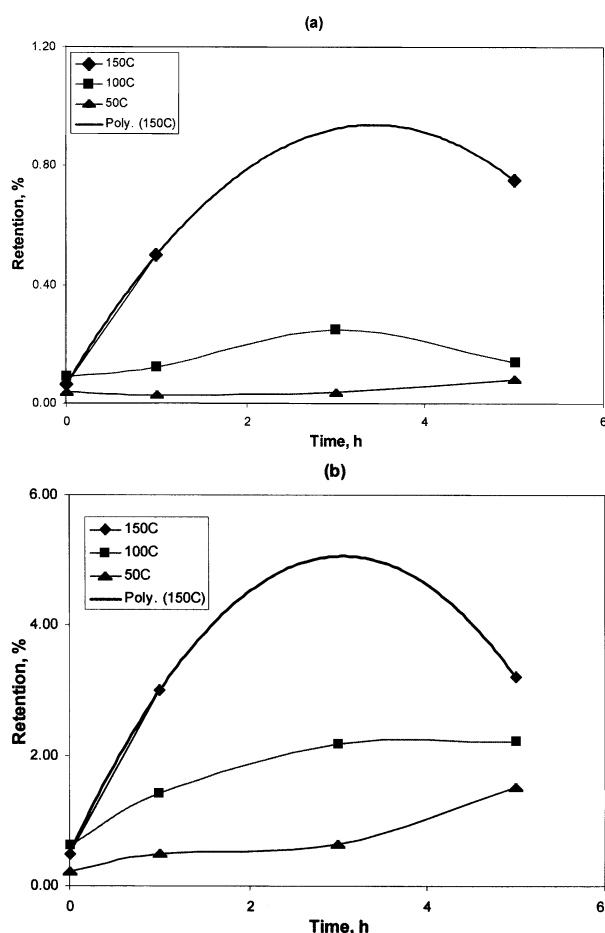


Fig. 6. Chromatographic paper type effect at different temperatures for $(C_5H_5)_3Sm$ using Whatman-1 (a) and Whatman-4 (b).

rapid “ligand condensation” as a very early prethermal competitive reaction, under prethermal term there are included reactions by direct collision, hot zone reactions, and epithermal reactions, but not thermal reactions occurring during the neutron irradiation [13].

Conclusion

The difference of thermal annealing behavior of the studied radioisotopes in the compounds under investigation could be the evidence for the difference of nuclear recoil reactions for each of them. The thermal annealing curves at 50°C were hardly changed. The thermal annealing was found to be affected by the method of separation. Isothermal curves could be explained by already known models. The differences in thermal annealing curves of the similar organometallic compounds could be related to the difference of electron configuration, recoil energy, lanthanide radii and the nature of the different lanthanides cations. As a result of this study, it was found that during the annealing processes in both $(C_{11}O_2H_{19})_3Ho$ and $(C_{11}O_2H_{19})_3Yb$ thermal reactions are involved, while in $(C_5H_5)_3Sm$ partially thermal reactions are involved, but $(C_5H_5)_3Ho$ did not involve any thermal reactions. As $(C_5H_5)_3Ho$ was

not much affected by thermal annealing, this could be related to that during annealing most of this compound was prethermally formed, while $(C_5H_5)_3Sm$ involved, in part, prethermal reactions. The use of different types of chromatographic paper gave different results for cyclopentadienyl compounds, but not for betadiketones, and this could be attributed to the different types of recoiled species. The thermal decomposition prevented the study of thermal annealing over 150°C for holmium compounds, and over 200°C for ytterbium and samarium compounds.

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