

A One-Pot Synthesis of “K(hfa) glyme” Adducts: Effect of the Polyether Length on the Ion Coordination Sphere

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Potassium complexes are starting to gather more and more interest from academia and industry because of their intriguing application possibilities. Novel adducts of potassium hexafluoroacetylacetonato [K(hfa)] with polyethers (monoglyme, diglyme, triglyme, and tetraglyme) were synthesized through a single step reaction and characterized through FT-IR spectroscopy as well as ¹H and ¹³C NMR spectroscopy. Single crystal X-ray diffraction studies enabled the identification of fascinating K coordination polymeric networks.

Introduction

In the late 1960s, the first report on cyclic polyethers, named crown ethers, was reported by Pedersen.^[1,2] This pioneering work opened the route to coordination chemistry of alkali ions to produce salt-polyether complexes relatively stable due to the ion-dipole interaction between the cation and the negatively charged O atoms of the polyether ring.^[3,4] Being the polyether neutral, an anion is always needed to counterbalance the cation positive charge and produce neutral complexes.^[5,6]

Among organic ligands, β-diketone is a mono-anion and belongs to the most exploited ligand systems for the complexation of metal ions.^[7-9] The presence of two oxygen atoms in the diketone offers a variety of metal-organic architectures behaving as bidentate ligands. Likewise, monoanionic bidentate N,N', of the type β-diketiminates^[10] and aminotroponimines,^[11] and N,O, such as azonaphthoxide^[12] are conventionally used ligands in the coordination chemistry of main group and transition metals.

Another essential issue in the synthesis of an alkali metal complex is the large ionic radius of the cation, which requires high coordination numbers. In fact, except for Li⁺, all the alkali metal ions from Na⁺ to Cs⁺ have an ionic radius larger than 1 Å, independently from the coordination number.^[13] Thus, the coordination sphere of the central ion in the traditional metal β-diketonates, is not saturated in case of s-block elements. This results into polymeric or oligomeric structures with low volatility.^[14] Among alkali metals, potassium compounds are actively studied for various applications such as precursors for the metalorganic chemical vapor deposition (CVD) processes of the ferroelectric (K,Na)NbO₃,^[15] the atomic layer deposition (ALD) of potassium oxide,^[16] sol-gel processes to produce hybrid organic-inorganic materials^[17] and in catalytic processes such as the ring-opening polymerization.^[18-20]

Potassium ion has a large ionic radius ($r_{6\text{-coord}} = 1.51 \text{ \AA}$),^[13] which is responsible for interesting coordination moieties giving rise, in function of the coordinating ligands to extended oligomeric or polymeric network.^[21-23]

Former studies have shown that glymes play a crucial role in stabilizing complexes of alkaline-earth,^[24-28] transition^[29,30] and rare-earth metals^[31,32] through oxygen-ion complexation. Recently, we have also devoted our attention to alkali metals, namely Li^[33] and Na.^[34] We have recently reported the synthesis and the crystal structures of Lithium β-diketonate complexes with a polymeric, [Li₂(hfa)₂·monoglyme·4H₂O]_n, and a dimer, [Li₂(hfa)₂·diglyme·2H₂O], structure.^[33] In regard to Na, a novel monomeric complex of the type, Na(hfa)·tetraglyme,^[34] has been synthesized and applied to the sol-gel processes of NaYF₄ phase.^[35] An analogous coordination architecture for Na has been observed in the heterobinuclear Na-RE (RE=Y and Gd) complexes of formula [RE(hfa)₄]⁻[Na·tetraglyme]⁺.^[36]

Here, we report on the synthesis of novel potassium complexes of the type “K(hfa)·glyme”, [Hhfa = 1,1,1,5,5,5-hexafluoro-2,4-pentanedione, glyme = monoglyme (1,2-dimethoxyethane), diglyme (bis(2-methoxyethyl)ether), triglyme (2,5,8,11-tetraoxadodecane) and tetraglyme (2,5,8,11,14-penta-oxapentadecane)], with glymes^[37] performing like crown ethers in terms of coordinating/solvating ions, i.e. in terms of chelating properties. Single crystal X-ray diffraction studies provide evidence of interesting coordination moieties. They have been also characterized by FT-IR spectroscopy, ¹H and ¹³C NMR.

The complexes are synthesized in a single step reaction, from the potassium hydroxide, Hhfa and glyme ligands in dichloromethane. A KOH:Hhfa:glyme stoichiometry of 1:1:1 was used for all the reactions. The mixture was refluxed under

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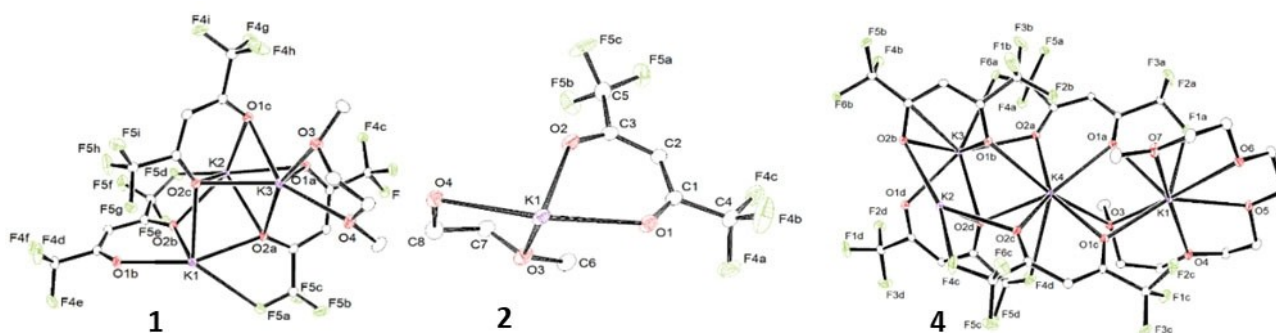


Figure 1. ORTEP-3 views of the asymmetric unit of **1**, **2** and **4**. For the sake of clarity hydrogen were not introduced and labels were added just for non-carbon atoms (ellipsoid probability = 20%).

stirring for 1 hour (Eq. 1). Adducts **1**, **2** and **4** are solid, while **3** is a colorless liquid at room temperature. All the adducts are soluble in ethanol, acetone, chloroform, and glymes.



1. $[\text{K}_3(\text{hfa})_3 \cdot \text{monoglyme}]_n$
2. $[\text{K}_2(\text{hfa})_2 \cdot \text{diglyme}]_n$
3. $\text{K}(\text{hfa}) \cdot \text{triglyme} \cdot \text{H}_2\text{O}$
4. $[\text{K}_4(\text{hfa})_4 \cdot \text{tetraglyme}]_n$

In Figure 1, ORTEP-3 representations of the asymmetric unit of the three compounds, **1**, **2** and **4** are reported, while the repeat units of **1**, **2** and **4** are reported in Figures S1, S2 and S3, respectively). Details of crystallographic data and refinement parameters are reported in Table S1. In all the three inves-

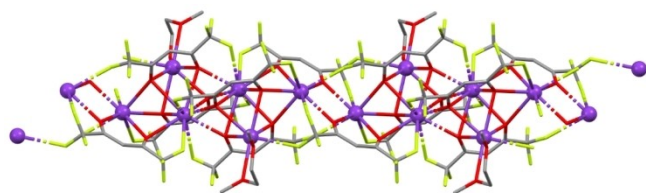


Figure 2. 1D polymer of adduct **1** (view along the ac plane diagonal).

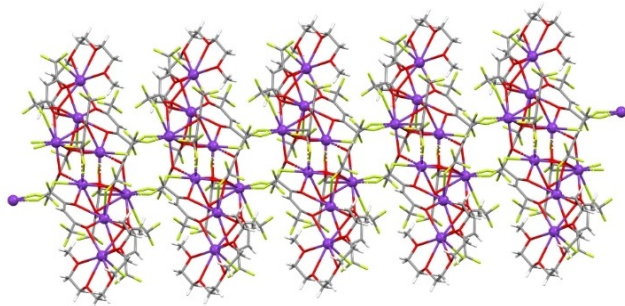


Figure 3. 1D polymer of adduct **2** (view along the b axis direction).

igated potassium complexes, due to the presence of bridging μ^2 and μ^3 oxygen atoms, 1D polymers are formed. In **1** they are directed along the b axis, while in **2** and **4** the propagation directions are along the c and a axis, respectively (Figure 2, Figure 3 and Figure 4). In all the three cases, no strong interactions connecting the polymer chains are present (Figure S4, Figure S5 and Figure S6). Concerning the disposition of the potassium cation inside the polymer chains, a sort of ladder is present, whose steps are formed by six and eight potassium ions, respectively, in **1** and **4**, (Figure S7), while in **2** the metal cations take a more linear disposition (Figure S7). **1** crystallizes in the monoclinic crystal system, $P2_1/n$ space group, and its asymmetric unit, the $[\text{K}_3(\text{hfa})_3 \cdot \text{monoglyme}]$, is reported in Figure 1. The three potassium cations, K1, K2 and K3 show a different coordination number (Figure S1). In all cases their coordination sphere is completed by fluorine atoms of a CF_3 moiety (see Table S2). More in details, K1 and K2 are coordinated by five hfa anions giving rise to a octa- and deca-coordinated environment. Finally, K3 is coordinated by three hfa anions and one monoglyme molecule, the resulting coordination is the hepta one. All the oxygen atoms of the hfa anions act as bridging donor atoms, while the monoglyme oxygen atoms bind just one potassium cation (K3).

Also **2** crystallizes in the monoclinic crystal system but this time the space group is $C2/c$. In this case the asymmetric unit is simpler than that observed in **1** and just one $[\text{K}(\text{hfa}) \cdot 0.5 \text{diglyme}]$ unit is present. The potassium cation K1 is hepta-coordinated by three hfa anion and one diglyme molecule (Figure S2).

The asymmetric unit of **4** (crystal system triclinic, space group P-1) contains the $[\text{K}_4(\text{hfa})_4 \cdot \text{tetraglyme}]$ unit. The four potassium cations show different coordination spheres (Figure S3). In fact, while K1, K3 and K4 are octa-coordinated (K1 by two

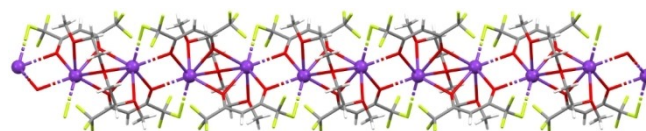


Figure 4. 1D polymer of adduct **4** (view along the b axis direction).

oxygen atoms and one fluorine atom of two hfa anion and by five oxygen atoms of a tetraglyme molecule, K3 by five oxygen and three fluorine atoms of four different hfa anions, and K4 by six oxygen and one fluorine atoms of four hfa anions and one oxygen atom of one tetraglyme molecule), K2 is hepta-coordinated by six oxygen and one fluorine atoms of five hfa anion.

Finally, the Cambridge Structural database (CSD, v 5.41, update 2 May 2020)^[38] was used to retrieve similar potassium complexes; just one structure was found containing a related CF₃-β-diketonate ligand (L in the following).^[39] Also in the case of the KL complex (CSD Refcode=COYPUE) a 1D polymer is formed. The K...K distances observed in **1**, **2** and **4** are comparable with those published for COYPUE (K...K distances range = 3.552–4.246 Å). Analogously, polymeric structures have been observed for Li^[40,41] and Na^[42] structures.

The FT-IR spectra of all the adducts **1–4** are reported in Figure S8. The absence of bands around 3500–3600 cm⁻¹ in the spectra of **1**, **2** and **4**, related to the stretching of O–H, is indicative of the lack of water molecules in the metal coordination sphere of **1**, **2** and **4**.

Comparing the FT-IR spectra of the adducts and that of the H-hfa ligand in the carbonyl range, it is possible to observe that the two peaks at about 1660 cm⁻¹ and 1530 cm⁻¹, associated with the C=O and C=C stretchings, respectively, are indicative of the hfa ligand coordination. The bands around 1350–1250 cm⁻¹ are due to the overlap of the vibration of stretching and/or bending of the polyether C–O bonds with the stretching vibration of the C–F bonds. The spectrum shows, at 863 cm⁻¹ and 1017 cm⁻¹, signals that can be considered as fingerprints of the glyme coordinated to the hexafluoroacetylacetonate potassium moiety. In addition, in the spectral range of 2800–3000 cm⁻¹, it is possible to find the C–H glyme stretching modes overlapped with Nujol features. The Nujol shows also peaks at 1461 and 1377 cm⁻¹.

The FT-IR spectrum of the adduct **3**, carried out on the neat liquid sample, is compared to the free Hhfa and triglyme ligands (Figure 5a). It shows a band around 3500 cm⁻¹, which is indicative of the presence of a water molecule coordinated to the metal ion. The shift of the signals related to the stretching of the C=O and C=C at 1675 and 1565 cm⁻¹, with respect to those characteristics of the free ligand observed at 1690 cm⁻¹ and 1630 cm⁻¹, dispels any doubts about the liquid nature of the obtained product confirming the hfa coordination to K (Figure 5b). The coordination of triglyme is confirmed as well due to the presence of the peak at about 2900 cm⁻¹, due to the C–H stretching of the polyether, and of the peaks at 855 cm⁻¹ and 1026 cm⁻¹ (Figure 5a and 5c).

The ¹H-NMR and ¹³C-NMR spectra of the “K(hfa)-glyme” adducts have been carried out dissolving **1** in CD₃COCD₃, while **2**, **3** and **4** in CDCl₃. All ¹H-NMR spectra show a singlet (5.51–5.71 δ) associated with the ring proton of the hfa ligand, while the glyme frameworks show different resonances. The spectrum of **1** shows two singlets for the methyl (δ=3.29) and the methylenic (δ=3.47) groups, an indication of a complete equivalence of the methylenic **b** protons (see Table 1 for the notation). For adduct **2**, in addition to the single resonance (δ =

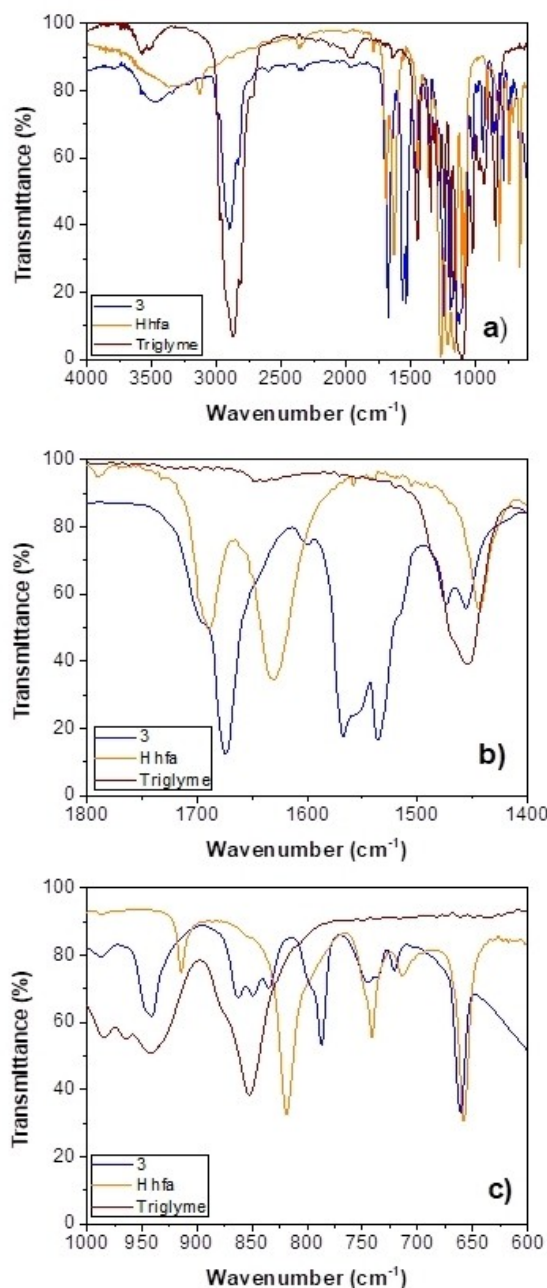


Figure 5. Comparison of the FT-IR spectra of the compound **3**, the Hhfa and the triglyme ligands: a) full 4000–600 cm⁻¹ region; b) C=O stretching region; and c) triglyme mode region.

3.30), associated with terminal methyl groups, bridging methylenic **b** and **c** protons give a multiplet structure centered at δ=3.53. In the adducts **3** and **4**, the methyl groups (**a** protons) are observed as a singlet at δ=3.30 and 3.35 ppm, respectively. The **b** and **c** protons are observed as a multiplet centered at δ=3.54 and a singlet for the **d** protons at δ=3.72 in the adducts **3**.

In adduct **4**, the **b** and **c** protons are observed as a multiplet centered at δ=3.57 and the protons **d** and **e** are associated with a multiplet at δ=3.63.

Table 1. ¹ H and ¹³ C NMR data of adducts 1–4.						
Complex	COCHCO	¹ H NMR		¹³ C NMR		
		polyether ^a	COCHCO	COCHCO	CF ₃	Polyether ^[a]
1	5.51 (s, 3 H)	a 3.29 (s, 6H) b 3.47 (s, 4H)	86.89 (s)	174.48 (q, ² J=32.2 Hz)	118.74 (q, ¹ J=287.5 Hz)	a 58.45 b 71.32
2	5.51 (s, 2 H)	a 3.30 (s, 6H) b, c 3.53 (m, 8H)	87.27 (s)	174.54 (q, ² J=32.4 Hz)	118.57 (q, ¹ J=287.3 Hz)	a 59.21 b, c 71.02
3	5.71 (s, 1H)	a 3.30 (s, 6H) b, c 3.54 (m, 8H) d 3.72 (s, 4H)	86.82 (s)	174.30 (q, ² J=32.9 Hz)	118.18 (q, ¹ J=287.4 Hz)	a 58.15 b 69.23 c, d 71.55
4	5.69 (s, 4 H)	a 3.35 (s, 6H) b, c 3.57 (m, 8H) d, e 3.63 (m, 8H)	86.21 (s)	174.29 (q, ² J=32.7 Hz)	118.35 (q, ¹ J=287.8 Hz)	a 59.85 b, c 69.73 d, e 71.37

[a] The following notation has been used from monoglyme (CH₃^a-O-CH₂^b)₂ through tetraglyme (CH₃^a-O-CH₂^b-CH₂^c-O-CH₂^d-CH₂^e-O).

The ¹³C NMR spectra (Table 1) can be assigned using comparative arguments with data of closely related complexes.^[25,26] Thus, resonances associated with the coordinated hfa ligands consist, in all cases, of singlets ($\delta \approx 90$) for the CH group, quartets ($\delta \approx 117$) for the CF₃ groups and quartets ($\delta \approx 176$) for the CO groups. The quartets are due to first order (CF₃; ¹J=286 Hz) and second order (CO; ²J=34 Hz) coupling with the CF₃ fluorine atoms. Specific glyme positions are reported in Table 1.

The thermal behaviors of the as-synthesized precursors have been investigated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) measurements. TGA experiments have been carried out under inert gas, such as N₂ (Figure 6), and under a reactant gas such as O₂ (Figure S9). TGA curves of the “K(hfa)₃glyme” 2 and 4, under N₂, show a single-step mass loss associated with the clean decomposition of the adducts, leaving residues of 34.1% and 23.2%, respectively. Adduct 1 shows, under N₂, a two-step mass-loss. The first weight loss of about 10.2% is in the range 25–95 °C, which accounts for the loss of the monoglyme of the [K₃(hfa)₃monoglyme] polymeric structure, the theoretical weight loss being equal to 10.8%. The main mass loss due to the clean adduct decomposition occurs in the range 218–271 °C, with a residue of 36.1%. For the liquid adduct 3, under

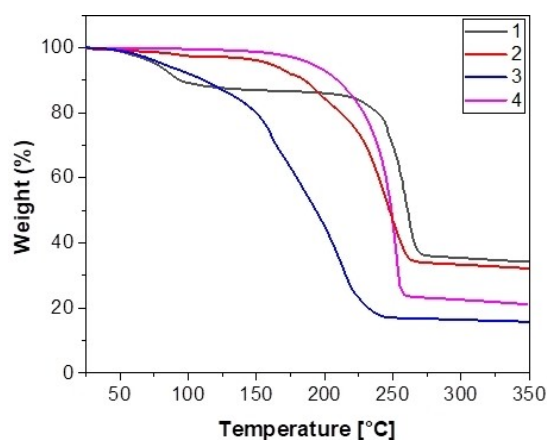


Figure 6. TGA curves of adducts 1–4 recorded under N₂.

N₂, a broad loss starts around 70 °C and completes at 270 °C, accounting for the H₂O molecule and organic component weight loss and leaving a residue of 16.5%. TG curves, recorded under O₂, have an analogous behavior (Figure S9), thus suggesting similar decomposition paths and a similar nature of the residues. The thermal analysis suggests thermal breakdown of the adducts, whose specific interpretation is not straightforward, but the energy dispersive X-ray analysis (EDX) of the residues gives an interesting feedback on the residue nature. The EDX spectra of the TGA curve residues of adduct 1, carried out under N₂ and under O₂, are reported in the Figures S10a and S10b, respectively. The EDX spectra show, in both cases, the presence of K and F peaks as main elements and traces of O.

Thus, the residue is in any case essentially formed by KF, but this finding is not surprising considering that fluoride phases of alkali and alkaline-earth metals are more stable than the respective oxides. On the other hand, this finding does not represent a drawback for their applications since it has been previously demonstrated that fluorinated precursors may be applied to the deposition of complex oxide phases, if the desired phase is thermodynamically more stable than the fluorides.^[43] Finally, even though these compounds leave an high residue, they can be applied in liquid-route processes, given their high solubility in common organic solvents.

Conclusion

In summary, the reported research provides a detailed study on the syntheses of four novel adducts of potassium. Various glymes have been used to complete the coordination sphere of the K ion, giving rise to stable compounds with interesting coordination moieties. The high solubility of the K adducts in common organic solvents makes them suitable for solution based methods to fabrication of thin films, either through liquid-assisted CVD or sol-gel/spin-coating approaches. The straightforward one-pot nature of the presently reported synthetic route envisages the possibility to easily synthesize and produce these compounds on large scale for industrial applications.

Deposition Numbers 2068746 (for 1), 2068747 (for 2), and 2068748 (for 4) contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

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Conflict of Interest

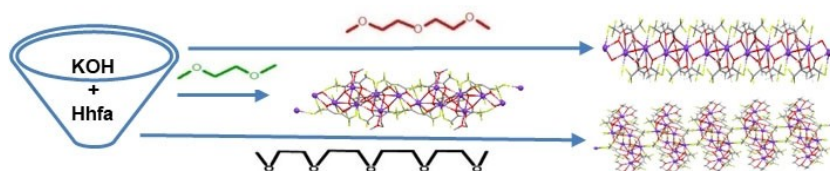
The authors declare no conflict of interest.

Keywords: Coordination chemistry · Glyme · Polymers · Potassium · Structure elucidation

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COMMUNICATIONS



A one-pot synthesis is applied to yield "K(hfa)glyme" complexes. The effect of glyme and fluorinated β -diketonate on the potassium's coordination and thermal properties are studied

through single crystal XRD, FT-IR, ^1H and ^{13}C NMR, TGA and DSC analyses. The complexes provide a viable option as K-precursors for sol-gel applications.

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1 – 6

A One-Pot Synthesis of "K(hfa)glyme" Adducts: Effect of the Polyether Length on the Ion Coordination Sphere

