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Non-covalent intramolecular Dy···F interactions in tris(pentafluorophenyl)methanolate dysprosium(III) complexes

Alexander N. Selikhov,[a,b] **Yulia V. Nelyubina**[b] **and Alexander A. Trifonov***[a,b]

[a] *G. A. Razuvaev Institute of Organometallic Chemistry, Russian Academy of Sciences, 603950 Nizhny Novgorod, Russian Federation. E-mail: trif@iomc.ras.ru*
[b] *A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 119334 Moscow, Russian Federation*

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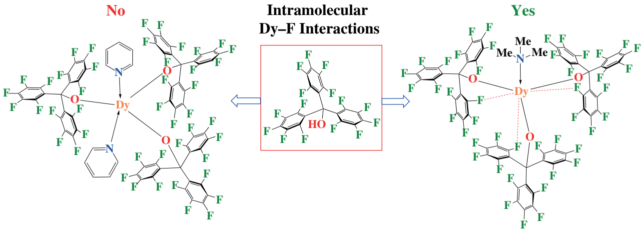
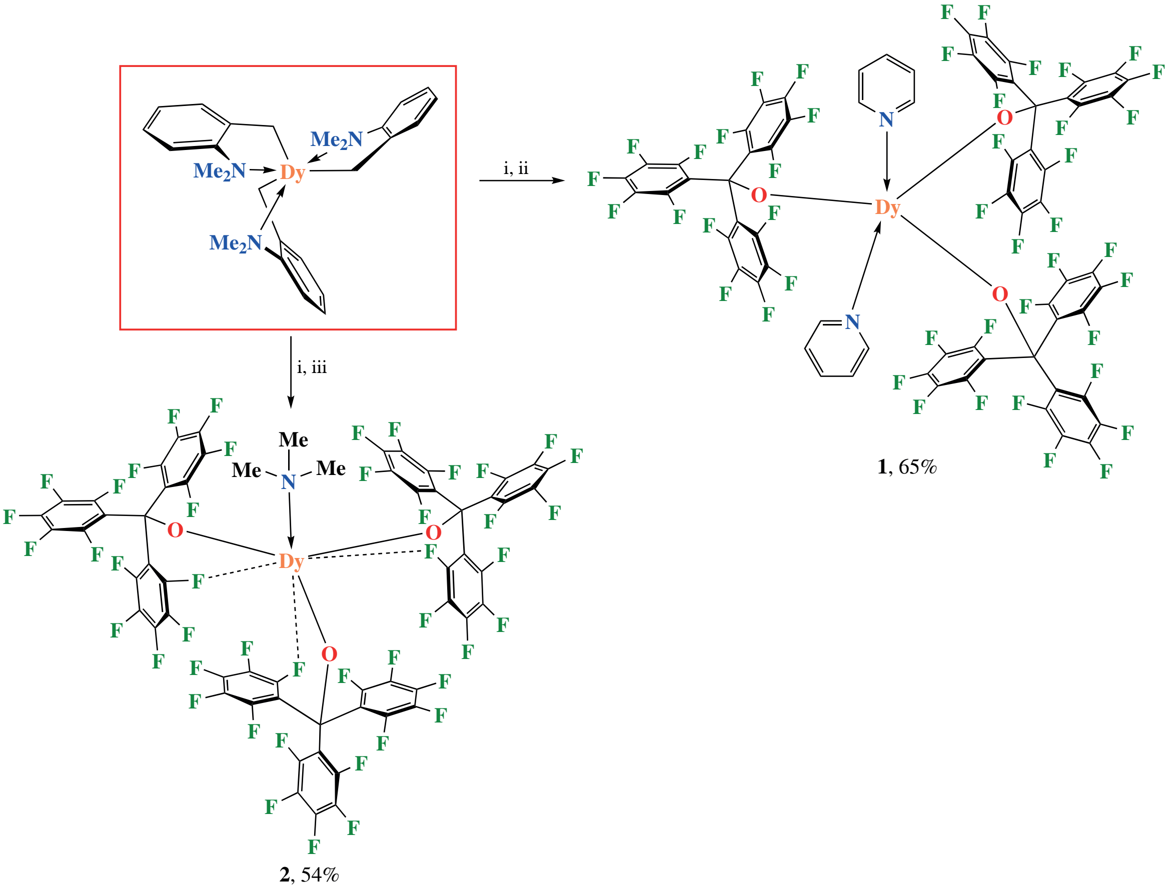
The reactions of $(C_6F_5)_3COH$ with (o-$Me_2NC_6H_4CH_2)_3Dy$ afford two new Dy^{3+} tris(alkoxy) complexes isolated in the form of pyridine and NMe_3 adducts $[(C_6F_5)_3CO]_3Dy(L)_n$ (L = py, n = 2 and L = Me_3N, n = 1). The first complex adopts trigonal bipyramidal geometry, while the second one is an almost ideal trigonal pyramidal. A characteristic feature of the latter formally four-coordinate complex is the presence of non-covalent intramolecular Dy···F interactions with each of $(C_6F_5)_3CO^-$ ligands reflected in short contacts, while in the former five-coordinate complex, such interactions were not detected.



Keywords: lanthanides, dysprosium, alkoxide anion, tris(pentafluorophenyl)methanolate, metal–fluorine interactions.

The chemistry of lanthanide complexes with fluorinated organic ligands represents a unique chapter among the diversity of organometallic derivatives due to the great promise of these complexes in the design of photo- and electroluminescent materials,[1–3] as well as advanced catalytic systems.[4,5] Dysprosium alkoxide complexes have been actively developed in the past decade to create novel high performant single ion magnets (SMMs, SIMs) and optical materials.[6,7] Special expectations in the development of new organometallic emitters are assigned to lanthanide complexes with perfluorinated ligands, since the replacement of hydrogens in alkoxide fragments by fluorine atoms improves the luminescent performance through suppressing the non-radiative deactivation process.[8,9] The pronounced electron withdrawing properties of perfluorinated organic anions would also affect the characteristics of slow relaxation of magnetization through the alteration of the lanthanide–ligand bonding.[10] Thus, the cationic Dy^{3+} complex with perfluorophenylethanolate ligands featuring axial symmetry exhibits a high anisotropy barrier of 1469 cm^{-1} and a blocking temperature of 22 K, making it one of the most performant alkoxide-based luminescent SMMs.[11] Dysprosium complexes with a terminal fluoride ligand demonstrate outstanding magnetic and luminescent characteristics.[12,13] Tris(perfluorophenyl)methanol $(C_6F_5)_3COH$, first prepared in 1967,[14] proved to be a unique ligand for constructing metal complexes possessing Lewis superacidity.[15] Its tris(perfluorophenyl)methanolate anion is distinguished by the presence of fifteen fluorine atoms, which gives it high acceptor properties and pronounced steric demand.[16,18] Moreover, the presence of fluorine sites is suitable for the formation of non-covalent M···F interactions,[19] stabilizing the metal complexes and providing a variety of coordination geometries. Thus, the homoleptic base-free aluminum tris-alkoxide $[(C_6F_5)_3CO]_3Al$ is the Lewis superacid with the highest fluoride ion affinity value (555 kJ mol^{-1}), and its aluminate $\{[(C_6F_5)_3CO]_3AlF\}^-$ can act as a weakly coordinating anion.[20] Application of the perfluorophenylethanolate anion also made it possible to synthesize a series of alkali metals, as well as magnesium, zinc, thallium and silver complexes featuring the presence of non-covalent M···F interactions.[21,22] Previously, we synthesized trigonal–pyramidal erbium complexes $[(C_6F_5)_3CO]_3Er(Base)$ that displayed a characteristic near infrared emission and slow relaxation of the magnetization under an applied static magnetic field, making them bifunctional luminescent SMMs.[23]

Apart from our research, no examples of lanthanide complexes containing ligand $(C_6F_5)_3CO^-$ are known to date. Herein, we report on the synthesis and characterization of two new dysprosium alkoxides with tris(perfluorophenyl)methanolate ligands isolated as pyridine and Me_3N adducts in which, depending on the degree of coordination unsaturation, non-covalent M···F interactions are present or absent. The reaction of $(C_6F_5)_3COH$ with (o-$Me_2NC_6H_4CH_2)_3Dy$ (3 : 1 molar ratio) was performed in toluene at room temperature within 2 h. After removal of all volatiles (toluene and o-$Me_2NC_6H_4CH_3$), the target complexes **1** and **2** were obtained as adducts with two molecules of pyridine (**1**) and one Me_3N molecule (**2**) after recrystallization of the solid residue from pyridine–hexane or Me_3N–heptane mixtures, respectively (Scheme 1). The dysprosium cation in the hypothetical tris-alkoxide $[(C_6F_5)_3CO]_3Dy$, due to the acceptor influence of the C_6F_5 substituents, has an extremely high Lewis acidity, and so it is not possible to trap and structurally characterize the solvent-free species.[23] This is in wide contrast to the published homoleptic solvent-free derivatives of aluminium,[20] magnesium and sodium,[22] which are attributed to monomeric $[(C_6F_5)_3CO]_3Al$, dimeric $\{[(C_6F_5)_3CO]_2Mg\}_2$ and tetrameric heterocubane



1, 65%

2, 54%

Scheme 1 *Reagents and conditions*: i, $(C_6F_5)_3COH$ (3 equiv.), toluene, room temperature, *o*-$Me_2NC_6H_4CH_3$; ii, recrystallization from pyridine–hexane mixture; iii, recrystallization from Me_3N–heptane mixture.

$[(C_6F_5)_3CONa]_4$ structures, respectively. Complexes **1** and **2** are quite stable in solvating solvents (THF, Py); unlike alkali metal derivatives, the nucleophilic ability of alkoxy RO^- anions is not sufficient for an attack at the *para*-position of the C_6F_5 rings. This correlates with the behavior in solution of fairly stable alkoxides $[(C_6F_5)_3CO]_nM$ (M = Mg, Zn, Ti).[22]

The structure of **1** and **2** (Figures 1 and 2) were confirmed by X-ray diffraction.[†] Their single crystals featured severely disordered molecules of pyridine and toluene used as solvents for recrystallization, and their contents were tentatively assigned to 1 and 1.5, respectively. Complex **1** adopts a distorted trigonal–bipyramidal geometry with three alkoxide ligands in equatorial positions and two pyridine donor molecules occupying the apical sites. The geometry of the Dy^{3+} center is similar to that found for complexes with bulky 2,6-diisopropylphenoxide ligands $(2,6\text{-}Pr^i_2C_6H_3O)_3Dy(NH_3)_2$ and $(2,6\text{-}Pr^i_2C_6H_3O)_3Dy(THF)_2$.[24] The Dy–$O_{alkoxide}$ bond lengths are in the narrow range of

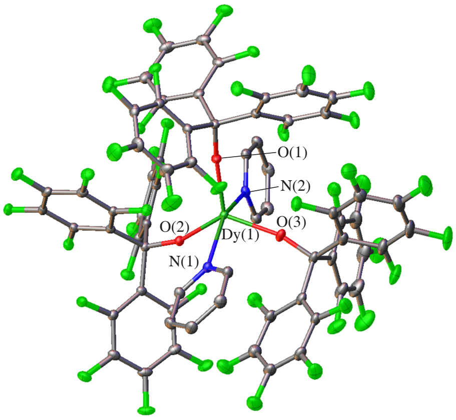


Figure 1 General view of complex **1** in representation of atoms *via* thermal ellipsoids at a 30% probability level. All hydrogen atoms are omitted, and labels are given only for the heteroatoms coordinated to the metal ion.

[†] *Crystal data for* $\mathbf{1}\cdot C_5H_5N$. $C_{72}H_{15}DyF_{45}N_3O_3$, M = 1987.37, monoclinic, space group $P2_1/n$, 100 K, a = 22.8913(4), b = 13.7991(3) and c = 24.0821(4) Å, β = 113.7100(10)°, V = 6964.9(2) Å^3, Z = 4, d_{calc} = 1.895 g cm^{-3}, F_{000} = 3852. Intensities of 79231 reflections were measured with a Bruker Quest D8 CMOS diffractometer [λ(MoKα) = 0.71073 Å, μ(MoKα) = 12.47 cm^{-1}, ω-scans, $2\theta < 54°$], and 15207 independent reflections (R_{int} = 0.0416) were used for the structure solution and refinement. Final R factors: R_1 = 0.0263 for 13050 observed reflections with $I > 2\sigma(I)$, wR_2 = 0.0632 and GOF = 1.032 for all the independent reflections.

Crystal data for $\mathbf{2}\cdot(C_6H_5CH_3)_{1.5}$. $C_{70.5}H_{21}DyF_{45}NO_3$, M = 1947.38, trigonal, space group $P3_1c$, 100 K, a = 15.6323(2) and c = 34.5221(6) Å, V = 7305.9(2) Å^3, Z = 4, d_{calc} = 1.770 g cm^{-3}, F_{000} = 3784. Intensities of 81431 reflections were measured with a Bruker Quest D8 CMOS diffractometer [λ(MoKα) = 0.71073 Å, μ(MoKα) = 11.86 cm^{-1}, ω-scans, $2\theta < 54°$], and 10229 independent reflections (R_{int} = 0.0586) were used for the structure solution and refinement. Final R factors: R_1 = 0.0771 for 9484 observed reflections with $I > 2\sigma(I)$, wR_2 = 0.2017 and GOF = 1.036 for all the independent reflections.

Using Olex2,[25] the structures were solved with the ShelXT[26] structure solution program using Intrinsic Phasing and refined with the XL[27] refinement package using the least-squares minimization. Positions of hydrogen atoms were calculated, and they were refined in the isotropic approximation within the riding model. Disordered lattice molecules of pyridine (1 mol) and toluene (1 mol) in **1** and **2**, respectively, were treated as diffuse contributions to the overall scattering without specific atom positions using the Solvent Mask routine implemented in OLEX2.

CCDC 2400659 (**1**) and 2400660 (**2**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* https://www.ccdc.cam.ac.uk.

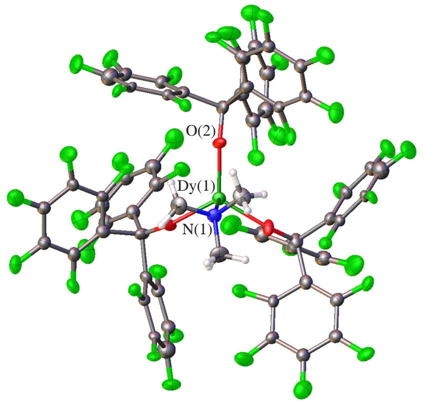


Figure 2 General view of **2** in representation of atoms *via* thermal ellipsoids at a 30% probability level. Only one symmetry-independent molecule is shown, the minor component of the disordered aryl rings is omitted, and labels are given only for the symmetry-independent heteroatoms coordinated to the metal ion. Each molecule of **2** occupies a special position, the three-fold axis.

2.0923(15)–2.1064(14) Å, and the Dy–N_{Py} coordination bonds are much longer [2.5156(19) and 2.5573(19) Å]. Due to the acceptor effect of perfluorinated substituents, the Dy–O bonds are found to be much shorter than those in complexes with non-fluorinated organic anions, for example, antracenolate complex $(C_{14}H_9O)_3Dy(Py)_3$ [Dy–O: 2.131(3)–2.164(3) Å],[28] neutral *ansa*-bridged bis(phenolate)chloride NHC complex [Dy–O: 2.129(7) and 2.152(8) Å] and cationic *ansa*-bridged bis(phenolate) NHC complex [Dy–O: 2.145(6) and 2.148(6) Å].[29] In contrast, they turned out to be close to the terminal Dy–O bond lengths in dysprosium alkoxide complexes coordinated by fluorinated ligands, $[(CF_3)_2CHO_2Dy(\mu_2\text{-}OHC(CF_3)_2)(H_2O)_2]_2$ and $[Dy(OCH(Me)C_6F_5)_2(THF)_5][BPh_4]$, which is quite reasonable.[11,30] Note that the N–Dy–N angle [153.70(6)°] is much less than 180°, leading to a deviation from linearity.

Complex **2** crystallizes with two symmetry-independent molecules, each occupying a special position, the three-fold axis that goes through the Dy–N bond. The coordination environment of the Dy^{3+} cation in **2** is composed of three oxygen atoms from alkoxide groups and one Me_3N molecule located in the apical position to provide an ideal trigonal–pyramidal geometry with the $O_{alkoxide}$–Dy–$O_{alkoxide}$ and O–Dy–N angles close to 120 and 90°, respectively. The Dy–$O_{alkoxide}$ bond lengths are 2.123(10) and 2.096(8) Å in two symmetry-independent molecules. Surprisingly, the dysprosium ion is located strictly in the plane formed by three alkoxide oxygen atoms located equatorially. Note a rather short Dy–N_{amine} bond [2.448(19) and 2.42(2) Å], which is not typical for dysprosium complexes with a coordinated terminal amine ligand.[31–35] This is obviously due to the high Lewis acidity of the dysprosium center, which causes a shift in electron density with a shortening of the coordination bond.[20] As a consequence, the coordination of the Me_3N to the metal center is quite rigid. The Dy^{3+} does not lose the amine upon redissolution of the complex in toluene (25 ºC), which does not occupy the vacant site.

A characteristic feature of previously published coordinatively unsaturated complexes with $(C_6F_5)_3CO^-$ anions is the presence of non-covalent M···F contacts.[20–23] Their number depends on the coordination environment of the metal ion in the complexes $[(C_6F_5)_3CO^-]M(L)$. As the previously published complexes of lithium,[21] aluminum[20] and erbium,[23] complex **2** features short contacts between *ortho*-fluorine atoms of the aromatic rings and the Dy^{3+} center. One of the three aryl rings of each $(C_6F_5)_3CO^-$ ligand is linked to the Dy^{3+} ion through short Dy···F_{ortho} contacts of 2.583 [2.43(5) Å for the minor component of the disordered aryl ring] and 2.611(9) Å in two symmetry-independent molecules. These distances are expected to lie below the sum of the van der Waals radii ($\Sigma^{Dy\cdots F}$ 4.29 Å),[36] but slightly greater than the sum of the covalent radii ($\Sigma_{cov}^{Dy\cdots F}$ 2.49 Å).[37] These distances are much shorter than those measured in cyclopentadienyl complexes $[\{Dy(Cp^{ttt})(Cp^*)\}\{Al\text{–}[OC(CF_3)_3]_4\}]$ with intermolecular Dy···F interactions 3.144(4) and 2.812(4) Å.[38] Thus, complex **2** has three short Dy···F contacts with each $(C_6F_5)_3CO^-$ ligand, which also contributes to the overall ideal geometry of the complex. Due to the coordination saturation and steric congestion of the dysprosium ion in the five-coordinate complex **1**, the smallest Dy···F distance is 2.8528(13) Å.

Despite the previously reported examples of lanthanide promoted C–F bond activation with LnF_3 elimination, complex **2** proved to be thermally stable and does not decompose when heated to 60 °C.[39–42] This may be due to the high steric shielding of the metal center, which gives an ideally rigid geometry, as well as the absence of O-containing coordinated bases.

In our ongoing work we plan to expand the range of alkoxide complexes of dysprosium with tris(pentafluorophenyl)methanolate ligands and to explore in detail their luminescent properties.

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Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi: 10.71267/mencom.7676.

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