Electronic supplementary materials *Mendeleev Commun.*, 2025, **35**, 303–306

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

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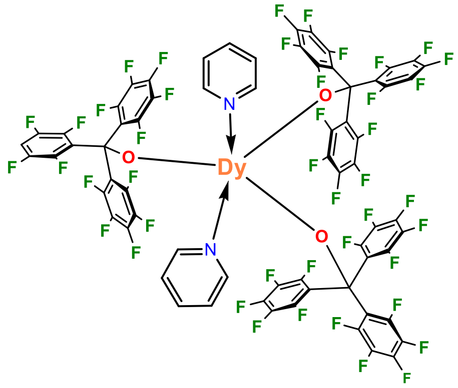
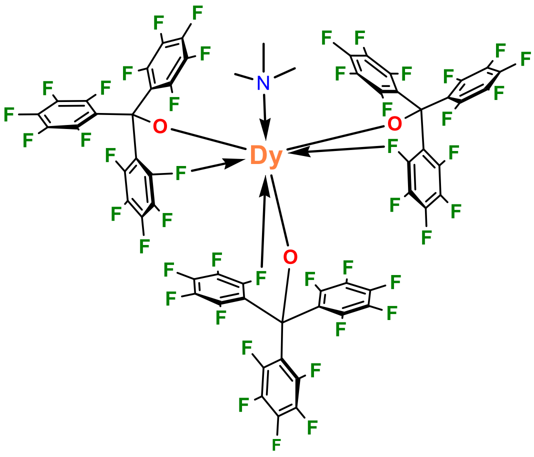
Experimental Section

General Procedures. All reactions were performed under a dry argon or nitrogen atmosphere using standard Schlenk techniques or under nitrogen atmosphere in a glovebox, unless indicated. Dry, oxygen-free solvents were used throughout. After being dried over KOH, THF and Et_2O were purified by distillation from sodium/benzophenone ketyl, hexane by distillation from sodium/potassium alloy prior to use. Pyridine and Me_3N were purchased from Aldrich, dried over CaH_2 and kept in an Ar-filled glovebox. $(C_6F_5)_3COH$ and $(o\text{-}Me_2NC_6H_4CH_2)_3Dy$ were prepared according to literature procedures.[S1],[S2] Another reagents and solvents were used as purchased from commercial suppliers. IR spectra were recorded as Nujol mulls or KBr plates on FSM 1201 and Bruker Vertex 70 instruments. The N, C, H elemental analyses were carried out in the microanalytical laboratory of the IOMC by means of a Carlo Erba Model 1106 elemental analyzer with an accepted tolerance of 0.4 unit on carbon (C), hydrogen (H), and nitrogen (N). Lanthanide metal analysis was carried out by complexometric titration.[S3]



Synthesis of tris[tris(pentafluorophenyl)methanolate]-dysprosium dipyridinate (1). $(C_6F_5)_3COH$ (0.50 g, 0.94 mmol) was added to a intensively stirred solution of $(o\text{-}Me_2NC_6H_4CH_2)_3Dy$ (0.17 g, 0.31 mmol) in toluene (5 ml) at -30 °C, and the mixture was slowly brought to room temperature. The yellow-green solution was stirred at this temperature for 24 hours, then all volatiles were removed in vacuum. The solid yellow-green residue was dried in vacuum for another 2 hours and then re-dissolved in boiling pyridine (3 ml). Complex **1** was obtained as colorless X-ray suitable crystals in 65% yield (0.39 g) by slow diffusion of hexane into the mother liquor in pyridine. IR (KBr): 1653 (s), 1526 (s), 1300 (s), 1264 (w), 1211 (w), 1123 (s), 1022 (s), 1005 (s), 991 (s), 978 (s), 864 (m), 845 (m), 798 (s), 767 (m), 747 (m), 700 (s), 665 (w), 638 (w), 621 (w), 571 (s). Anal. Calcd for $C_{67}H_{10}DyF_{45}N_2O_3$ (1908.26 g/mol): C 42.17; H 0.53; N, 1.47; Dy, 8.52. Found: C 42.43; H 0.60; N, 1.34; Dy, 8.41.



Synthesis of tris[tris(pentafluorophenyl)methanolate]-dysprosium trimethylaminate (2). $(C_6F_5)_3COH$ (0.50 g, 0.94 mmol) was added to a intensively stirred solution of (*o*-$Me_2NC_6H_4$ $CH_2)_3Dy$ (0.17 g, 0.31 mmol) in toluene (5 ml) at -30 °C, and the mixture was slowly brought to room temperature. The yellow-green solution was stirred at this temperature for 24 hours, then all volatiles were removed in vacuum. The solid yellow-green residue was dried in vacuum for another 2 hours and then re-dissolved in fresh boiling toluene (10 ml). Trimethylamine (0.10 g, 1.80 mmol) was added to the solution slowly. Complex **2** was obtained as colorless X-ray suitable crystals in 54% yield (0.31 g) by slow concentration of the mother liquor in toluene. IR (KBr): 1653 (s), 1527 (s), 1301 (s), 1264 (w), 1245 (w), 1210 (w), 1125 (s), 1015 (s), 1005 (s), 991 (s), 978 (s), 864 (m), 845 (m), 840 (m), 798 (s), 767 (m), 747 (m), 700 (s), 665 (w), 631 (w), 620 (w), 570 (s). Anal. Calcd for $C_{60}H_9DyF_{45}NO_3$ (1809.16 g/mol): C 39.83; H 0.50; N, 0.77; Dy, 8.98. Found: C 39.98; H 0.55; N, 0.76; Dy, 8.78.

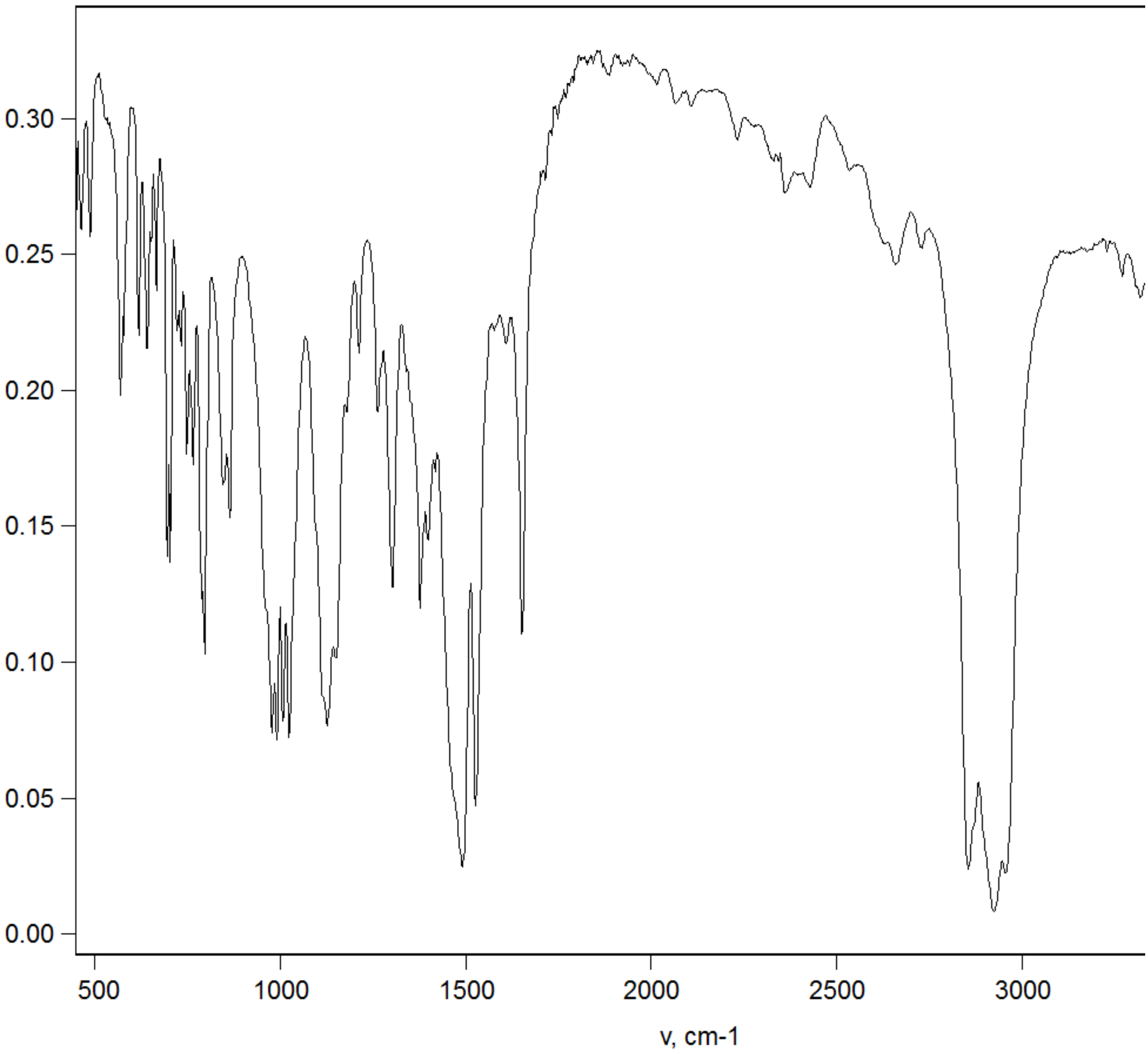


Figure S1. IR (KBr) spectrum of **tris[tris(pentafluorophenyl)methanolate]dysprosium dipyridinate (1)**.

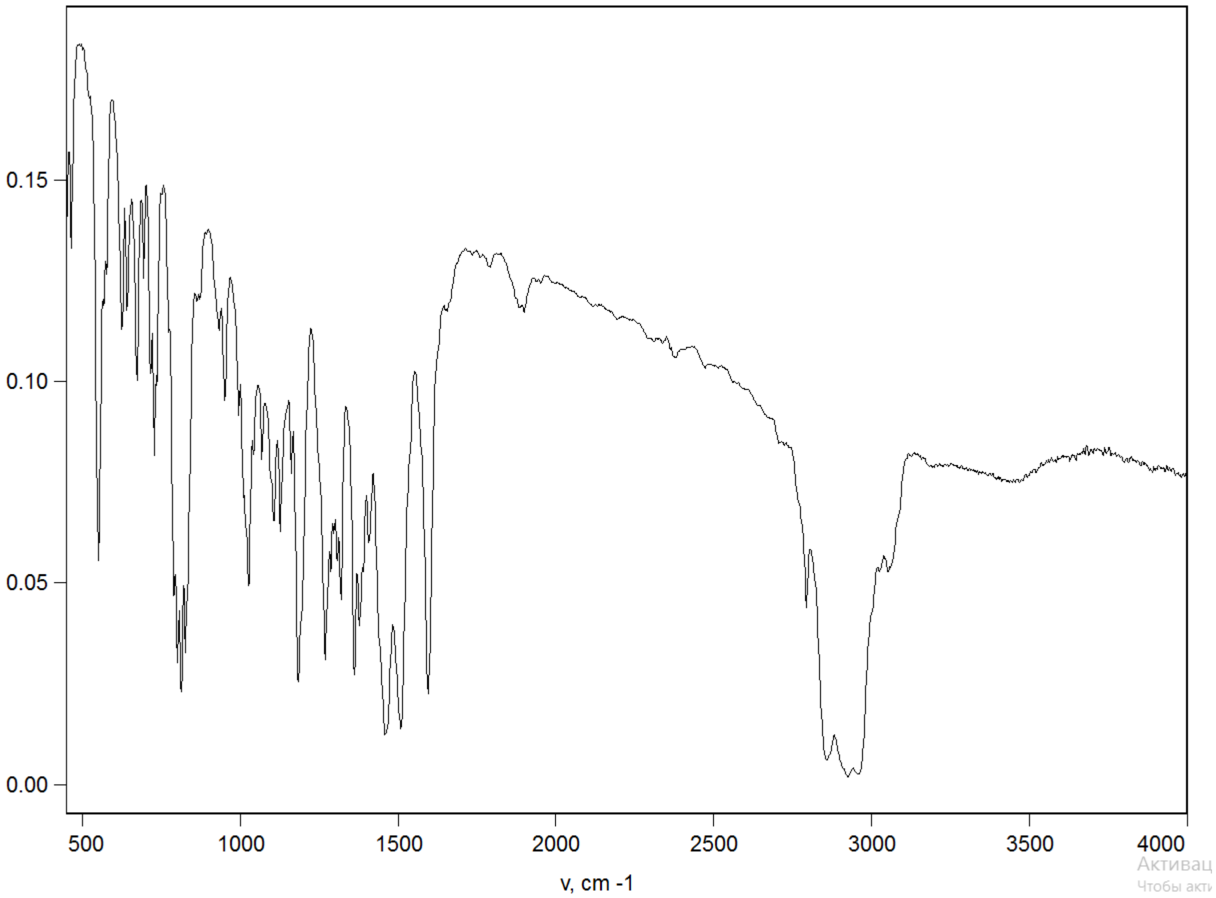


Figure S2. IR (KBr) spectrum of **tris[tris(pentafluorophenyl)methanolate]dysprosium trimethylaminate (2)**.

References

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S3. S. J. Lyle and Md. M. Rahman, *Talanta*, 1963, **10**, 1177; https://doi.org/10.1016/0039-9140(63)80170-8.