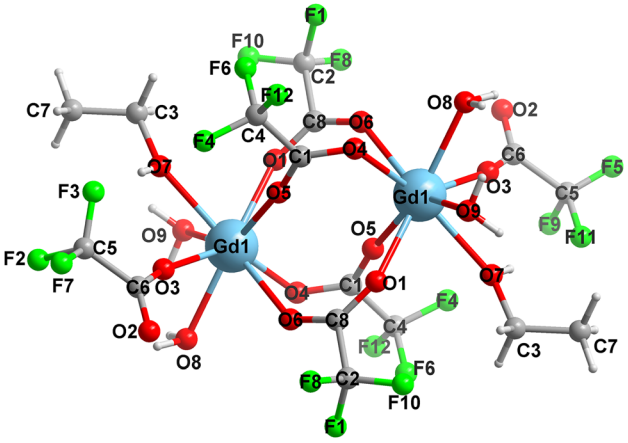
Bo-Kai Ling* and Ling-Yu Huang

Crystal structure of tetraaqua-bis(ethanol-κO)-tetrakis(μ$_2$-trifluoroacetate-κ^{2}O:O′)-bis(trifluoroacetate-κ^{2}O)digadolinium(III) $Gd_2C_{16}H_{20}O_{18}F_{18}$



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Abstract

$Gd_2C_{16}H_{20}O_{18}F_{18}$, triclinic, $P\bar{1}$ (no. 2), a = 8.652(3) Å, b = 9.204(4) Å, c = 12.305(5) Å, α = 78.156(5)°, β = 72.483(5)°, γ = 62.797(4)°, V = 828.4(6) Å^3, Z = 1, $R_{gt}(F)$ = 0.0227, $wR_{ref}(F^2)$ = 0.0554, T = 150 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.20 × 0.10 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	4.14 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	28.9°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9812, 3894, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3639
$N(param)_{refined}$:	277
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

1 Source of materials

362 mg Gd_2O_3 (1 mmol) was placed in a 30 ml scintillation flask and 10 ml ethanol was added. With stirring, trifluoroacetic acid was dropwise added until the solution is clear. Then the solution was stirred at room temperature for 8 h. Thereafter, the solution was filtered and stood for evaporation. Colorless block-like crystals were isolated for three days (yield 30 %, based on Gd).

2 Experimental details

Absorption corrections were used by using multi-scan program [1]. Using Olex2 [2], the structure was solved with the ShelXT [3] structure solution program and refined with the ShelXL [4] refinement package. The H atoms were fixed, fixed U_{iso} were set to 1.2 times of all C(H) groups.

3 Comment

Rare-earth elements are widely used in computer hard drives, electric and hybrid vehicles, flat-screen monitors and televisions and defense application. Furthermore, rare-earth salts are selected as precursor for synthesizing more complicated rare-earth compounds [5–7]. In this work, we report the single crystal structure of gadolinium trifluoroacetate dimer, which provides a resource for further synthesis.

***Corresponding author: Bo-Kai Ling**, Key Laboratory of Special Functional and Smart Polymer Materials of Ministry of Industry and Information Technology, Xi'an Key Laboratory of Functional Organic Porous Materials, School of Chemistry and Chemical Engineering, Northwestern Polytechnical University, Xi'an, Shaanxi 710072, P.R. China, E-mail: bruceling001@163.com
Ling-Yu Huang, T&R Office of Physics and Chemistry, Army Academy of Border and Coastal Defence, Xi'an, Shaanxi, 710000, P.R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6442 (4)	0.3216 (4)	0.1567 (3)	0.0188 (6)
C2	0.0806 (5)	0.3660 (4)	0.1701 (3)	0.0285 (8)
C3	0.0967 (6)	0.6616 (7)	0.3773 (4)	0.0494 (11)
H3A	0.1412	0.5453	0.4077	0.059*
H3B	0.0020	0.6835	0.3388	0.059*
C4	0.6402 (5)	0.2822 (4)	0.2845 (3)	0.0275 (7)
C5	0.5541 (5)	0.8140 (5)	0.3652 (3)	0.0345 (9)
C6	0.5986 (4)	0.8182 (4)	0.2336 (3)	0.0227 (7)
C7	0.0197 (7)	0.7649 (9)	0.4720 (5)	0.0726 (19)
H7A	−0.0273	0.8804	0.4428	0.109*
H7B	0.1121	0.7421	0.5116	0.109*
H7C	−0.0777	0.7417	0.5255	0.109*
C8	0.2359 (4)	0.4129 (4)	0.1036 (3)	0.0185 (6)
F1	0.1403 (3)	0.2190 (3)	0.2282 (2)	0.0429 (6)
F2[a]	0.4048 (8)	0.9602 (7)	0.3958 (4)	0.0723 (15)
F3[a]	0.4976 (11)	0.7046 (8)	0.4166 (5)	0.066 (2)
F4	0.6304 (4)	0.4043 (3)	0.3320 (2)	0.0482 (6)
F5[b]	0.562 (2)	0.9211 (15)	0.4042 (9)	0.076 (3)
F6	0.4957 (4)	0.2565 (4)	0.3388 (2)	0.0541 (7)
F7[a]	0.6711 (8)	0.8206 (13)	0.4045 (5)	0.088 (3)
F8	−0.0016 (3)	0.3541 (3)	0.0987 (3)	0.0507 (7)
F9[b]	0.6874 (12)	0.6652 (11)	0.4047 (6)	0.056 (2)
F10	−0.0402 (4)	0.4715 (3)	0.2451 (3)	0.0609 (9)
F11[b]	0.4147 (15)	0.797 (2)	0.4208 (10)	0.078 (5)
F12	0.7822 (4)	0.1496 (3)	0.3058 (2)	0.0562 (8)
Gd1	0.35011 (2)	0.73221 (2)	0.08943 (2)	0.01450 (5)
O1	0.2078 (3)	0.5559 (3)	0.1091 (2)	0.0211 (5)
O2	0.7304 (3)	0.8436 (4)	0.1801 (2)	0.0347 (6)
O3	0.4915 (3)	0.7977 (3)	0.1957 (2)	0.0262 (5)
O4	0.7278 (3)	0.2042 (3)	0.0954 (2)	0.0226 (5)
O5	0.5569 (3)	0.4664 (3)	0.1261 (2)	0.0247 (5)
O6	0.3706 (3)	0.2993 (3)	0.05281 (19)	0.0194 (4)
O7	0.2412 (3)	0.6877 (4)	0.2946 (2)	0.0343 (6)
H7	0.311 (4)	0.690 (6)	0.3289 (14)	0.051*
O8	0.3083 (3)	1.0093 (3)	0.0275 (2)	0.0221 (5)
H8A	0.3423	1.0434	0.0712	0.033*
H8B	0.1970	1.0710	0.0368	0.033*
O9	0.0336 (3)	0.8966 (3)	0.1285 (2)	0.0289 (6)
H9A	0.0126	0.9949	0.1017	0.043*
H9B	−0.0076	0.9026	0.2004	0.043*

[a]Occupancy: 0.65, [b]Occupancy: 0.35.

This compound is composed of six trifluoroacetate ligands, two ethanol, four water molecules and two Gd metal atoms. The gadolinium center is octahedrally coordinated by two oxygen atoms of bridging trifluoroacetate ligands and four oxygen atoms from two water molecules, one trifluoroacetate ligand and one ethanol molecule. The distances of Gd–O bonds are in range of 2.328–2.451 Å [8].

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