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The crystal structure of N,N′-carbonylbis(2,6-difluorobenzamide), $C_{15}H_8F_4N_2O_3$

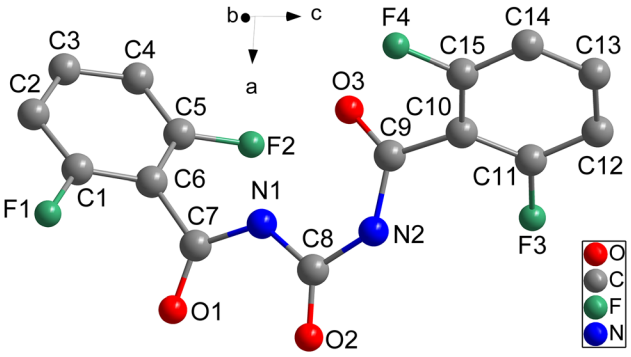

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.10 × 0.10 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.14 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7562, 2538, 0.017
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2036
$N(param)_{refined}$:	217
Programs:	Bruker,[1] Olex2,[2] Shelx[3,4]

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Abstract

$C_{15}H_8F_4N_2O_3$, monoclinic, $P2_1/n$ (no. 14), a = 11.2759(7) Å, b = 9.0182(5) Å, c = 14.2235(8) Å, β = 93.834(2)°, V = 1443.13(15) Å^3, Z = 4, $R_{gt}(F)$ = 0.0391, $wR_{ref}(F^2)$ = 0.1067, T = 273.15 K.

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The molecular structure is shown in the figure (hydrogen atoms are omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

2,6-Difluorobenzamide (2 mol, 31.424 g) and oxalyl chloride (1 mol, 12.693 g) were suspension in xylene (200 g). The suspension liquid was stirred at 110 °C for 5 h. After the reaction, the temperature was lowered to 0 °C and filtered to obtain white solid. The white solid is N,N′-carbonylbis(2,6-difluorobenzamide). The title crystals were obtained by slow evaporation from mixed solvent (tetrahydrofuran: acetonitrile = 1: 2) at room temperature.

2 Experimental details

Absorption corrections were performed by using multi-scan program.[1] The structure was solved with Shelx.[3,4] Hydrogen atoms were placed in their geometrically idealized positions. Hydrogen atoms were constrained to ride on their parent atoms.

3 Comment

Urea and urea derivatives have been deeply studied in various fields and achieved good results, many have even been used in our lives.[5,6] Urea and urea derivatives can be doped with TiO_2 and other inorganic salts to form a variety of nanocatalysts, which are used for catalytic reactions in various fields.[7,8] The nano-photocatalyst containing urea and TiO_2 can effectively photocatalyze the degradation of Rhodamine B (RhB) in the environment to protect the environment.[9]

The title molecule in the crystal structure is a flexible molecule. Each title molecule contains two difluorobenzoyl

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.71482 (10)	0.70170 (14)	0.60608 (9)	0.0524 (3)
C1	0.49112 (17)	0.7604 (2)	0.48237 (12)	0.0512 (5)
F1	0.56506 (12)	0.67886 (17)	0.43223 (8)	0.0825 (4)
N1	0.58291 (12)	0.57846 (17)	0.69289 (10)	0.0495 (4)
H1	0.508481	0.571010	0.701878	0.059*
N2	0.60112 (12)	0.39048 (18)	0.80418 (10)	0.0485 (4)
H2	0.646167	0.328640	0.835708	0.058*
C2	0.40149 (19)	0.8385 (2)	0.43555 (14)	0.0632 (5)
H2A	0.391379	0.836163	0.370139	0.076*
O2	0.76563 (11)	0.48702 (19)	0.74570 (10)	0.0682 (4)
F2	0.44690 (12)	0.84624 (17)	0.72227 (8)	0.0805 (4)
O3	0.40635 (10)	0.45465 (16)	0.77544 (9)	0.0624 (4)
C3	0.32682 (18)	0.9205 (2)	0.48753 (15)	0.0608 (5)
H3	0.265691	0.974556	0.456643	0.073*
F3	0.57854 (12)	0.37325 (15)	1.00662 (8)	0.0754 (4)
C4	0.34052 (17)	0.9244 (2)	0.58395 (15)	0.0577 (5)
H4	0.289978	0.980576	0.618827	0.069*
F4	0.31656 (14)	0.15866 (17)	0.77712 (12)	0.1001 (5)
C5	0.43123 (16)	0.8429 (2)	0.62745 (12)	0.0485 (4)
C6	0.51026 (14)	0.75930 (18)	0.57957 (11)	0.0405 (4)
C7	0.61406 (14)	0.67950 (18)	0.62686 (11)	0.0400 (4)
C8	0.65917 (15)	0.4877 (2)	0.74625 (11)	0.0472 (4)
C9	0.48175 (14)	0.3800 (2)	0.81779 (11)	0.0438 (4)
C10	0.44847 (14)	0.26979 (19)	0.88941 (12)	0.0438 (4)
C11	0.49159 (18)	0.2741 (2)	0.98228 (13)	0.0533 (5)
C12	0.4490 (3)	0.1849 (3)	1.05044 (17)	0.0830 (8)
H12	0.478343	0.192337	1.112933	0.100*
C13	0.3618 (3)	0.0845 (3)	1.0238 (3)	0.1026 (12)
H13	0.331960	0.023036	1.069106	0.123*
C14	0.3179 (2)	0.0725 (3)	0.9324 (3)	0.0946 (10)
H14	0.260151	0.002131	0.915093	0.113*
C15	0.36021 (18)	0.1657 (2)	0.86689 (18)	0.0644 (6)

groups and one urea groups. The C–C distances are in the range of 1.364(5)–1.495(2) Å. The C–N distances are in the range of 1.371(2)–1.396(2) Å.[10] The C–O distances are in the range of 1.201(2)–1.213(2) Å. The C–F distances are in the range of 1.339(3)–1.355(2) Å.[11] The title molecule was assembled into a twisted structure by four hydrogen bonds and four $\pi\cdots\pi$ interactions. First hydrogen bond is N2–H2–O1, the distance of N2–H2 bond is 0.86 Å, the distance of H2–O1 bond is 2.07 Å, the angle of N2–H2–O1 is 167°. Second hydrogen bond is C12–H12–F2, the distance of C12–H12 bond is 0.93 Å, the distance of H12–F2 bond is 2.46 Å, the angle of C12–H12–F2 is 168°.[12] Third hydrogen bond is C13–H13–O1, the distance of C13–H13 bond is 0.93 Å, the distance of H13–O1 bond is 2.49 Å, the angle of C13–H13–O1 is 148°. Fourth hydrogen bond is N1–H1–O3, the distance of N1–H1 bond is 0.86 Å, the distance of H1–O3 bond is 1.92 Å, the angle of N1–H1–O3 is 139°, this hydrogen bond is a intramolecular hydrogen bond.[13]

First $\pi\cdots\pi$-interaction present in Cg1 (the centroid of ring C1–C6) and Cg1^{i} (symmetry code *i*: 1 – *x*, 2 – *y*, 1 – *z*). The shortest centre-to-centre distance of Cg1 and Cg1^{i} is 3.5828(11) Å. Second $\pi\cdots\pi$-interaction present in Cg1 and Cg2^{ii} (the centroid of ring C10–C15: symmetry code *ii*: 1/2 – *x*, 1/2 + *y*, 3/2 – *z*). The shortest centre-to-centre distance of Cg1 and Cg2^{ii} is 3.9347(13) Å. Third $\pi\cdots\pi$-interaction present in Cg2 and Cg1^{iii} (symmetry code *iii*: 1/2 – *x*, –1/2 + *y*, 3/2 – *z*). The shortest centre-to-centre distance of Cg2 and Cg1^{iii} is 3.9347(13) Å. Fourth $\pi\cdots\pi$-interaction present in Cg2 and Cg2^{iv} (symmetry code *iv*: 1 – *x*, –*y*, 2 – *z*). The shortest centre-to-centre distance of Cg2 and Cg2^{iv} is 3.9636(14) Å.[14]

All bond lengths are generally in the expected ranges.[15]

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