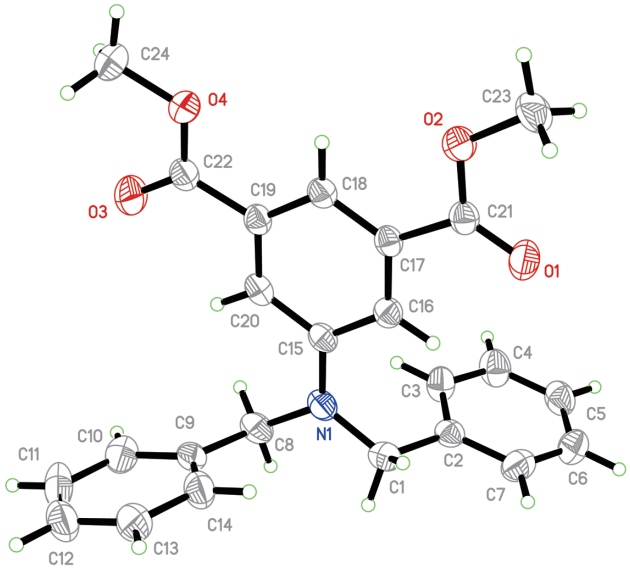
Open Access

Yaping Li, Dajun Sun*, Julia Ming and Guanfang Su

Crystal structure of dimethyl 5-(dibenzylamino) isophthalate, $C_{24}H_{23}NO_4$



DOI 10.1515/ncrs-2016-0043
Received February 1, 2016; accepted May 10, 2016; available online May 31, 2016

Abstract

$C_{24}H_{23}NO_4$, monoclinic, $P2_1/n$ (no. 14), $a = 13.492(6)$ Å, $b = 9.259(4)$ Å, $c = 15.993(8)$ Å, $\beta = 94.009(9)°$, $V = 1993.0(16)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0409$, $wR_{ref}(F^2) = 0.1201$, $T = 293(2)$ K.

CCDC no.: 1479167

The asymmetric unit of the crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Dajun Sun,** Department of Vascular Surgery, The China-Japan Union Hospital of Jilin University, Changchun 130033, P. R. China, e-mail: drsundj@163.com
Yaping Li and Guanfang Su: Department of Ophthalmology, The Second Hospital of Jilin University, Changchun 130041, P. R. China
Julia Ming: St Erik's Eye Hospital, Karolinska Institutet, Polhemsgatan 50, SE-112 82, Stockholm, Sweden

Table 1: Data collection and handling.

Crystal:	Colorless blocks
	Size 0.27 × 0.25 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.9 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω
$2\theta_{max}$, completeness:	52.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10556, 4037, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2639
$N(param)_{refined}$:	263
Programs:	SHELX [6], Bruker programs [7]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.05525(13)	−0.1785(2)	−0.06012(11)	0.0562(5)
H1A	0.0078	−0.2165	−0.1029	0.067*
H1B	0.0295	−0.0871	−0.0415	0.067*
C2	0.06177(13)	−0.28157(18)	0.01288(10)	0.0483(4)
C3	0.13981(15)	−0.3746(2)	0.02618(12)	0.0615(5)
H3	0.1916	−0.3717	−0.0092	0.074*
C4	0.14317(17)	−0.4725(2)	0.09099(13)	0.0732(6)
H4	0.1968	−0.5353	0.0988	0.088*
C5	0.06814(17)	−0.4779(2)	0.14378(12)	0.0687(6)
H5	0.0705	−0.5437	0.1878	0.082*
C6	−0.01012(17)	−0.3863(3)	0.13148(13)	0.0749(6)
H6	−0.0617	−0.3896	0.1671	0.090*
C7	−0.01338(15)	−0.2884(2)	0.06654(13)	0.0685(6)
H7	−0.0672	−0.2259	0.0589	0.082*
C8	0.16092(15)	−0.2091(2)	−0.18044(10)	0.0580(5)
H8A	0.1148	−0.2886	−0.1908	0.070*
H8B	0.2276	−0.2480	−0.1812	0.070*
C9	0.14514(13)	−0.1015(2)	−0.25111(10)	0.0515(4)
C10	0.18728(16)	−0.1264(2)	−0.32525(12)	0.0680(6)
H10	0.2294	−0.2051	−0.3299	0.082*
C11	0.1680(2)	−0.0363(3)	−0.39290(13)	0.0875(8)
H11	0.1958	−0.0560	−0.4433	0.105*
C12	0.1085(2)	0.0815(3)	−0.38650(14)	0.0863(8)
H12	0.0954	0.1420	−0.4324	0.104*
C13	0.06851(16)	0.1099(3)	−0.31302(14)	0.0805(7)
H13	0.0292	0.1915	−0.3080	0.097*
C14	0.08589(15)	0.0185(2)	−0.24575(12)	0.0691(6)
H14	0.0571	0.0382	−0.1958	0.083*
C15	0.21798(13)	−0.06017(17)	−0.05978(10)	0.0446(4)
C16	0.20603(12)	0.00105(17)	0.01858(10)	0.0456(4)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H16	0.1503	−0.0225	0.0469	0.055*
C17	0.27545(12)	0.09607(17)	0.05507(9)	0.0429(4)
C18	0.36014(12)	0.13144(18)	0.01569(9)	0.0461(4)
H18	0.4069	0.1949	0.0405	0.055*
C19	0.37354(12)	0.07000(18)	−0.06150(10)	0.0452(4)
C20	0.30450(13)	−0.02349(18)	−0.09831(10)	0.0476(4)
H20	0.3155	−0.0634	−0.1502	0.057*
C21	0.25400(13)	0.16115(18)	0.13664(10)	0.0467(4)
C22	0.45971(13)	0.1075(2)	−0.11017(10)	0.0511(4)
C23	0.30982(16)	0.3226(2)	0.24368(11)	0.0694(6)
H23A	0.3653	0.3848	0.2583	0.104*
H23B	0.3043	0.2518	0.2869	0.104*
H23C	0.2500	0.3790	0.2379	0.104*
C24	0.59744(14)	0.2593(3)	−0.11894(13)	0.0776(6)
H24A	0.6405	0.1803	−0.1305	0.116*
H24B	0.6343	0.3310	−0.0865	0.116*
H24C	0.5712	0.3012	−0.1708	0.116*
N1	0.14804(11)	−0.15165(15)	−0.09763(8)	0.0528(4)
O1	0.18006(10)	0.13789(15)	0.17213(8)	0.0672(4)
O2	0.32461(9)	0.25123(14)	0.16560(7)	0.0608(4)
O3	0.47438(11)	0.05655(17)	−0.17674(8)	0.0777(4)
O4	0.51747(9)	0.20784(15)	−0.07289(7)	0.0600(4)

Source of material

5-(dibenzylamino)isophthalic acid (0.2 mmol, 0.072 g) was added to 20 mL methanol. The mixture was stirred and refluxed for twenty minutes. The mixture was filtered and colorless block crystals were collected after evaporation of the methanol after 3 days.

Experimental details

The C-bound H atoms were positioned with idealized geometry and were refined with $U_{iso}(H) = 1.2U_{eq}(C)$ using a riding model with C—H = 0.93 Å for aromatic H atoms, C—H = 0.96 Å for methyl H atoms and C—H = 0.97 Å for methylene H atoms.

Discussion

In recent years, the rational design and assembly of metal organic frameworks (MOFs) have received remarkable attention in order to develop new functional materials with various potential applications [1, 2]. It is well known that the flexible branched multicarboxylate ligand are excellent building blocks for the construction of MOFs [3–5]. If we can synthesize multidentate ligand as higher-connected vertices, higher-connected frameworks can be expected. Taking these into account, we design and synthesize a new multidentate carboxylic acid ligand. The title compound is a derivative of this carboxylic acid. In the crystal structure of the title compound, the bond lengths and angles are within expected ranges. The dihedral angle of the benzene ring with two ester groups to the other two benzene rings are 87.10° and 86.11°, respectively. The dihedral angle between the two benzene rings is 27.06°. The π−π interactions between adjacent benzene rings provide additional stability of the crystal packing, with a closest distance between benzene mean planes of 3.701(2) Å.

Acknowledgements: The project were supported by the Pharmaceutical industry Promotion Project of Jilin Province (20140311015YY).

References

1. Song, X. Z.; Song, S. Y.; Zhao, S. N.; Hao, Z. M.; Zhu, M.; Meng, X.; Wu, L. L.; Zhang, H. J.: Single-crystal-to-single-crystal transformation of a europium(III) metal-organic framework producing a multi-responsive luminescent sensor. Adv. Funct. Mater. **24** (2014) 4034–4041.
2. Murray, L. J.; Dinca, M.; Long, J. R.: Hydrogen storage in metal-organic frameworks. Chem. Soc. Rev. **38** (2009) 1294–1314.
3. He, J. Y.; Yao, Q. G.; Liu, J. J.; Jia, H. Z.; Yu, H. W.: Crystal structure of dimethyl 4-(4-chlorophenyl)-1,4-dihydropyridine-3,5-dicarboxylate, $C_{15}H_{14}ClNO_4$. Z. Kristallogr. NCS **229** (2014) 123–124.
4. Zhu, M.; Song, X. Z.; Song, S. Y.; Meng, X.; Zhao, S. N.; Wu, L. L.; Wang, C.; Zhang, H. J.: A temperature-responsive smart europium metal-organic framework switch for reversible capture and release of intrinsic Eu^{3+} ions. Adv. Sci. **2** (2015) 1500012.
5. Zhao, S. N.; Li, L. J.; Song, X. Z.; Zhu, M.; Hao, Z. M.; Meng, X.; Wu, L. L.; Feng, J.; Song, S. Y.; Wang, C.; Zhang, H. J.: Lanthanide ion codoped emitters for tailoring emission trajectory and temperature sensing. Adv. Funct. Mater. **25** (2015) 1463–1469.
6. Sheldrick, G. M.: Ashort history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
7. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA, 2009.