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Crystal structure of 4,4'-(1,3,5,7-tetraoxo-3a,4,4a,5,7,7a,8,8a-octahydro-4,8-ethenopyrrolo [3,4-*f*]isoindole-2,6(1*H*,3*H*)-diyl)dibenzoic acid, $C_{26}H_{18}N_2O_8$

DOI 10.1515/ncrs-2016-0042

Received February 1, 2016; accepted May 23, 2016; available online June 1, 2016

Abstract

$C_{26}H_{18}N_2O_8$, triclinic, $P\bar{1}$ (no. 2), $a = 7.7679(19)$ Å, $b = 11.000(3)$ Å, $c = 13.370(3)$ Å, $\alpha = 75.682(5)^\circ$, $\beta = 81.286(5)^\circ$, $\gamma = 69.905(5)^\circ$, $V = 1036.7(5)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0643$, $wR_{\text{ref}}(F^2) = 0.1680$, $T = 293(2)$ K.

CCDC no.: 1481276

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.

Source of material

4,4'-(1,3,5,7-tetraoxo-3a,4,4a,5,7,7a,8,8a-octahydro-4,8-ethenopyrrolo [3,4-*f*]isoindole-2,6(1*H*,3*H*)-diyl)dibenzoic acid (0.2 mmol, 0.098 g), 20 mL methanol, and 10 mL acetone were mixed. The mixture was stirred and refluxed for twenty minutes. After filtration, the clear solution was left to slowly evaporate in air to give colorless block crystals of the title compound suitable for X-ray diffraction.

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Table 1: Data collection and handling.

Crystal:	Colorless blocks
Wavelength:	Size $0.23 \times 0.20 \times 0.18$ mm
μ :	Mo K_α radiation (0.71073 Å)
Diffractometer, scan mode:	1.2 cm^{-1}
$2\theta_{\text{max}}$, completeness:	Bruker SMART, φ and ω
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	52.2° , >99%
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	6593, 4091, 0.058
$N(\text{param})_{\text{refined}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2215
Programs:	325
	SHELX [8], Bruker programs [9]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6806(5)	0.4154(3)	0.3255(3)	0.0183(8)
C2	0.7337(5)	0.2994(3)	0.2744(3)	0.0177(8)
H2	0.7295	0.2195	0.3259	0.021*
C3	0.9788(5)	0.3929(4)	0.2534(3)	0.0198(8)
C4	0.9316(5)	0.2828(3)	0.2271(3)	0.0163(8)
H4	1.0141	0.1969	0.2599	0.020*
C5	0.6055(5)	0.3260(4)	0.1872(3)	0.0191(8)
H5	0.4765	0.3432	0.2136	0.023*
C6	0.9418(5)	0.2927(3)	0.1096(3)	0.0176(8)
H6	1.0663	0.2851	0.0778	0.021*
C7	0.6788(5)	0.2015(4)	0.1396(3)	0.0168(8)
H7	0.6672	0.1244	0.1922	0.020*
C8	0.8814(5)	0.1768(3)	0.0995(3)	0.0160(8)
H8	0.9570	0.0943	0.1411	0.019*
C9	0.5805(5)	0.2146(3)	0.0463(3)	0.0179(8)
C10	0.8922(5)	0.1607(3)	−0.0092(3)	0.0165(8)
C11	0.6338(5)	0.4391(4)	0.1049(3)	0.0206(9)
H11	0.5380	0.5165	0.0844	0.025*
C12	0.8049(5)	0.4215(4)	0.0632(3)	0.0202(9)
H12	0.8371	0.4838	0.0097	0.024*
C13	0.6738(5)	0.1567(4)	−0.1260(3)	0.0177(8)
C14	0.6540(5)	0.2473(4)	−0.2186(3)	0.0200(8)
H14	0.6633	0.3305	−0.2228	0.024*
C15	0.6198(5)	0.2142(4)	−0.3061(3)	0.0210(9)
H15	0.6052	0.2753	−0.3688	0.025*

Table 2 (continued)

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C16	0.6077(5)	0.0897(4)	−0.2989(3)	0.0175(8)
C17	0.6308(5)	−0.0020(4)	−0.2051(3)	0.0203(9)
H17	0.6242	−0.0860	−0.2010	0.024*
C18	0.6635(5)	0.0310(4)	−0.1183(3)	0.0200(8)
H18	0.6783	−0.0300	−0.0555	0.024*
C19	0.5652(5)	0.0500(4)	−0.3875(3)	0.0201(8)
C20	0.8327(5)	0.5757(4)	0.3460(3)	0.0206(9)
C21	0.8807(5)	0.6759(4)	0.2755(3)	0.0232(9)
H21	0.8977	0.6753	0.2052	0.028*
C22	0.7968(5)	0.5778(4)	0.4499(3)	0.0207(9)
H22	0.7560	0.5129	0.4957	0.025*
C23	0.8226(5)	0.6788(3)	0.4851(3)	0.0200(8)
H23	0.8016	0.6806	0.5552	0.024*
C24	0.8797(5)	0.7768(3)	0.4161(3)	0.0179(8)
C25	0.9033(5)	0.7771(4)	0.3107(3)	0.0217(9)
H25	0.9343	0.8456	0.2639	0.026*
C26	0.9264(5)	0.8741(4)	0.4546(3)	0.0182(8)
N1	0.8289(4)	0.4648(3)	0.3097(2)	0.0191(7)
N2	0.7138(4)	0.1851(3)	−0.0348(2)	0.0174(7)
O1	0.4183(3)	0.2445(2)	0.03844(19)	0.0224(6)
O2	1.0260(3)	0.1324(2)	−0.06777(19)	0.0232(6)
O3	0.5368(3)	0.4631(2)	0.37074(19)	0.0229(6)
O4	1.1215(4)	0.4173(3)	0.2295(2)	0.0296(7)
O5	0.5904(3)	0.1232(2)	−0.47795(18)	0.0226(6)
H5A	0.5643	0.0952	−0.5231	0.034*
O6	0.5093(4)	−0.0453(3)	−0.37520(19)	0.0256(6)
O7	0.9483(3)	0.9754(2)	0.38415(19)	0.0249(6)
H7A	0.9737	1.0252	0.4118	0.037*
O8	0.9476(4)	0.8603(2)	0.54649(19)	0.0240(6)

Experimental details

The C-bound H atoms were positioned with idealized geometry and were refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ using a riding model with C–H = 0.93 Å for aromatic H atoms and C–H = 0.98 Å for methine H atoms. The O-bound H atoms were positioned with idealized geometry and were refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ using a riding model with O–H = 0.82 Å.

Discussion

It is well known that carboxylates are excellent building blocks for the construction of coordination polymers because the carboxylate groups may induce core aggregation and link these discrete clusters into an extended framework by virtue of its bridging ability [1–7]. Taking these into account, we design and synthesize a new carboxylic acid as starting material for such syntheses.

This compound may be a good candidate for artificial eye vitreous. The values of the geometric parameters of the title structure are within normal ranges and experimental errors. The benzene rings make dihedral angles of 54.79(10)° and 82.30 (11)°, respectively, with the five-member rings they are attached to. In the crystal structure, molecules are linked via pairs of O–H···O hydrogen bonds between the hydroxy group and the carbonyl O atom, which may be effective in the stabilization of the structure.

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

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