Crystal structure of 2,2′-(cyclohexane-1,2-diyl)diisoindolin-1-one, $C_{22}H_{22}N_2O_2$

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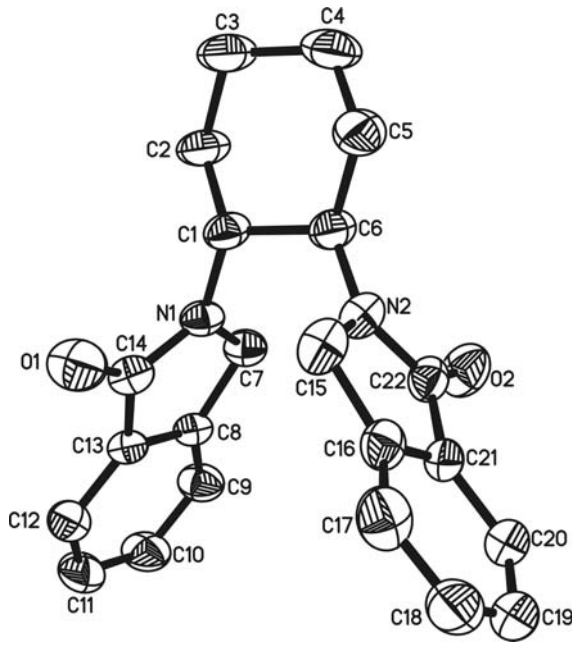

Abstract

$C_{22}H_{22}N_2O_2$, monoclinic, $P2_1/n$ (no. 14), a = 11.7852(4) Å, b = 10.1276(4) Å, c = 15.4341(7) Å, β = 92.278(4)°, V = 1840.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.081, T = 293 K.

Source of material

A mixture of 2-formylbenzoic acid (3.02 g, 0.02 mol), cyclohexane-1,2-diamine(1.14 g, 0.01 mol) and methanol (100 mL) was stirred and refluxed for an hour, then the mixture was cooled to room temperature, and sodium borohydride (1.52 g, 0.04 mol) was added. After stirring for an hour, the solvent was evaporated. The residue was dissolved in 100 mL water, then concentrated HCl was added drop by drop. A white precipitate – 2,2′-(cyclohexane-1,2-diylbis(azanediyl))bis(methylene)dibenzoic acid (H_2L) – was obtained (yield 63 %). The title compound was prepared from a mixture of $Cd(OH)_2$ (0.015 g, 0.1 mmol), 1,4-di(1*H*-imidazol-1-yl)butane (0.019 g, 0.1 mmol) and H_2L (0.038 g, 0.1 mmol) in 8 mL methanol. The mixture was heated in a sealed Teflon-lined steel autoclave at 80 °C for three days. After the mixture had been cooled to room temperature at a rate of 10 °C/h, colorless crystals of the title compoud were obtained (yield 24 %).

The title compound was an unexpected product. We were attempting to obtain a complex contain both 1,4-di(1*H*-imidazol-1-yl)butane and Cd(II) cation, but failed. What happened was intramolecular condensation of 2,2′-(cyclohexane-1,2-diylbis-(azanediyl))-bis(methylene)dibenzoic acid.

Experimental details

All the H atoms on C atoms were placed in calculated positions with d(C—H) = 0.93 - 0.98 Å, and U_{iso}(H) = 1.2 U_{eq}(C). The weak crystal diffraction leading to a low $N_{gt}/N(parameter)$ ratio. The structure was checked by PLATON, there are no additional symmetry elements found.

Discussion

The title crystal structure exhibits discrete molecules. The two C=O distances are 1.228 Å and 1.229 Å, respectively, and the C—N bond distances are in the range 1.346 - 1.463 Å. The molecules are interconnected to 2D layers by the C–H···O interactions: d(C1···O1) = 2.860 Å, d(C6···O2) = 2.859 Å, d(C7···O1) = 3.225 Å, d(C10···O2) = 3.271 Å, d(C17···O1) = 3.386 Å and the angles are ∠C1–H1–O1 = 105.8°, ∠C6–H6–O2 = 106.4°, ∠C7–H7B–O1 = 152.1°, ∠C10–H10–O2 = 137.6° and ∠C17–H17–O1 = 155.7°, respectively.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.15 × 0.16 × 0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.81 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7944, 4234
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1700
$N(param)_{refined}$:	235
Programs:	SHELXS-97, SHELXL-97 [1]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(19)	4*e*	0.4823	0.6370	0.3712	0.118
H(20)	4*e*	0.4097	0.4259	0.3656	0.096
H(3A)	4*e*	–0.1146	0.0358	0.6907	0.125
H(3B)	4*e*	–0.0834	0.1807	0.7187	0.125
H(7A)	4*e*	0.3100	0.0681	0.5627	0.074
H(7B)	4*e*	0.2717	–0.0395	0.6299	0.074
H(4A)	4*e*	–0.1746	0.1874	0.5835	0.140
H(4B)	4*e*	–0.0879	0.0861	0.5469	0.140
H(18)	4*e*	0.4295	0.7798	0.4764	0.124
H(6)	4*e*	0.1158	0.1567	0.5322	0.080

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(15A)	4*e*	0.2225	0.4545	0.6460	0.089
H(15B)	4*e*	0.1222	0.5109	0.5859	0.089
H(1)	4*e*	0.1209	0.2581	0.7018	0.076
H(12)	4*e*	0.5386	0.2201	0.8211	0.092
H(9)	4*e*	0.5159	–0.0966	0.6030	0.088
H(2A)	4*e*	0.0743	0.0474	0.7473	0.102
H(2B)	4*e*	0.0705	–0.0037	0.6513	0.102
H(5A)	4*e*	–0.0325	0.3476	0.5992	0.113
H(5B)	4*e*	–0.0375	0.2980	0.5027	0.113
H(17)	4*e*	0.3022	0.7173	0.5801	0.108
H(11)	4*e*	0.6954	0.0934	0.7903	0.105
H(10)	4*e*	0.6833	–0.0630	0.6832	0.100

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(19)	4*e*	0.4308(2)	0.6098(3)	0.4118(1)	0.085(1)	0.133(2)	0.078(2)	–0.031(1)	–0.008(1)	0.033(2)
N(1)	4*e*	0.2542(1)	0.1479(1)	0.67381(9)	0.0423(7)	0.0573(8)	0.0699(9)	0.0047(7)	0.0074(7)	0.0042(7)
C(20)	4*e*	0.3876(1)	0.4850(2)	0.4078(1)	0.072(1)	0.105(2)	0.064(1)	–0.013(1)	0.0016(9)	0.015(1)
N(2)	4*e*	0.1816(1)	0.3359(1)	0.54436(8)	0.0684(8)	0.0586(8)	0.0640(8)	0.0056(7)	0.0160(7)	0.0064(7)
O(1)	4*e*	0.29751(9)	0.2885(1)	0.78653(9)	0.0814(8)	0.0942(9)	0.117(1)	0.0081(7)	0.0155(7)	–0.0409(9)
C(3)	4*e*	–0.0675(1)	0.1112(2)	0.6777(1)	0.049(1)	0.137(2)	0.128(2)	0.000(1)	0.012(1)	0.054(2)
C(7)	4*e*	0.3091(1)	0.0451(2)	0.6237(1)	0.0502(9)	0.065(1)	0.071(1)	0.0039(8)	0.0056(8)	–0.0015(9)
O(2)	4*e*	0.2568(1)	0.2244(1)	0.43091(8)	0.1103(9)	0.0871(9)	0.0767(9)	–0.0060(7)	0.0251(7)	–0.0147(8)
C(22)	4*e*	0.2495(1)	0.3236(2)	0.4761(1)	0.065(1)	0.071(1)	0.052(1)	0.0085(9)	0.0068(9)	0.002(1)
C(4)	4*e*	–0.0961(1)	0.1585(2)	0.5873(2)	0.049(1)	0.162(2)	0.138(2)	–0.004(1)	–0.004(1)	0.044(2)
C(8)	4*e*	0.4269(1)	0.0438(2)	0.6648(1)	0.0426(8)	0.0541(9)	0.061(1)	–0.0025(7)	0.0074(7)	0.0027(8)
C(18)	4*e*	0.3991(2)	0.6951(2)	0.4749(2)	0.106(2)	0.094(2)	0.107(2)	–0.036(1)	–0.026(1)	0.037(2)
C(14)	4*e*	0.3238(1)	0.2020(2)	0.7353(1)	0.057(1)	0.059(1)	0.071(1)	–0.0026(9)	0.0130(9)	–0.001(1)
C(13)	4*e*	0.4343(1)	0.1364(2)	0.7292(1)	0.0442(9)	0.062(1)	0.059(1)	–0.0068(8)	0.0073(8)	0.0024(9)
C(6)	4*e*	0.1055(1)	0.2318(2)	0.5712(1)	0.0528(9)	0.073(1)	0.075(1)	0.0022(9)	0.0083(8)	0.016(1)
C(21)	4*e*	0.3102(1)	0.4489(2)	0.4682(1)	0.0546(9)	0.074(1)	0.051(1)	–0.0025(9)	0.0008(8)	0.0129(9)
C(15)	4*e*	0.1939(1)	0.4638(2)	0.5865(1)	0.090(1)	0.064(1)	0.071(1)	0.017(1)	0.018(1)	0.006(1)
C(1)	4*e*	0.1344(1)	0.1843(2)	0.6625(1)	0.0450(9)	0.071(1)	0.075(1)	0.0071(8)	0.0129(8)	0.0158(9)
C(12)	4*e*	0.5344(1)	0.1569(2)	0.7774(1)	0.066(1)	0.095(1)	0.069(1)	–0.011(1)	0.005(1)	–0.013(1)
C(9)	4*e*	0.5202(1)	–0.0330(2)	0.6465(1)	0.056(1)	0.076(1)	0.089(1)	0.0020(9)	0.008(1)	–0.016(1)
C(16)	4*e*	0.2784(1)	0.5328(2)	0.5319(1)	0.073(1)	0.061(1)	0.061(1)	0.0045(9)	–0.0006(9)	0.012(1)
C(2)	4*e*	0.0568(1)	0.0723(2)	0.6876(1)	0.0466(9)	0.104(1)	0.106(1)	0.000(1)	0.0136(9)	0.037(1)
C(5)	4*e*	–0.0193(1)	0.2722(2)	0.5622(1)	0.061(1)	0.121(2)	0.100(2)	0.018(1)	0.003(1)	0.039(1)
C(17)	4*e*	0.3227(2)	0.6589(2)	0.5370(1)	0.115(2)	0.067(1)	0.087(1)	–0.003(1)	–0.011(1)	0.007(1)
C(11)	4*e*	0.6272(1)	0.0812(2)	0.7590(1)	0.051(1)	0.124(2)	0.088(1)	0.003(1)	–0.003(1)	–0.008(1)
C(10)	4*e*	0.6198(1)	–0.0124(2)	0.6946(1)	0.050(1)	0.100(2)	0.101(2)	0.013(1)	0.007(1)	–0.009(1)

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