Crystal structure of diethyl 1-(cyclohexyl carbamoyl)-3-methyl-6-nitro-1*H*-indene-1,2-dicarboxylate, $C_{23}H_{28}N_2O_7$

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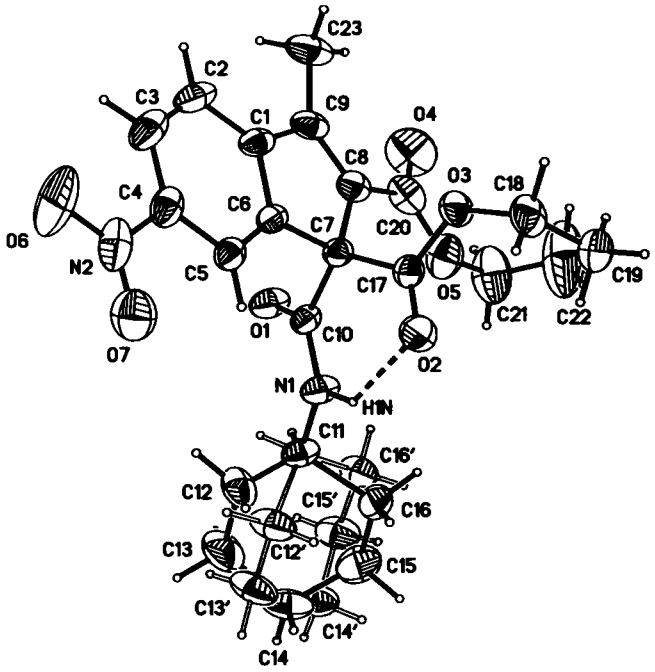

Abstract

$C_{23}H_{28}N_2O_7$, triclinic, $P\bar{1}$ (no. 2), $a = 9.200(2)$ Å, $b = 10.844(2)$ Å, $c = 12.300(2)$ Å, $\alpha = 82.660(3)°$, $\beta = 82.260(4)°$, $\gamma = 71.730(4)°$, $V = 1149.9$ Å^3, $Z = 2$, $R_{gt}(F) = 0.055$, $wR_{ref}(F^2) = 0.109$, $T = 293$ K.

Source of material

The title compound was synthesized by the reaction of cyclohexyl isocyanide and diethyl acetylene dicarboxylate with 1-(4-nitrophenyl)ethanone in *p*-xylene, then purified by column chromatography on silica-gel using a mixture of ethyl acetate and *n*-hexane (*v*/*v* 40:60) as eluent. Recrystallization from ethanol gave crystals suitable for X-ray structure analysis.

Discussion

Some of substituted indene derivatives have been shown to exhibit biological activity [1]. There are some reports on the crystal structures of indene derivatives [2-5]. The title molecule crystallized centrosymmetrically with one molecule per asymmetric unit. The indene moiety is nearly planar and the cyclohexyl ring exhibits complete chair conformation. The molecule is stabilized by one intramolecular N–H···O hydrogen bond. Details of the N1–H1A···O2 bond are: d(N1—H1A) = 0.94 Å, d(H1A···O2) = 2.02 Å, d(N1···O2) = 2.735 Å, ∠N1–H1A···O2 = 132.0°.

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Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.60 × 0.50 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.95 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω-$^5/_3\theta$
$2\theta_{max}$:	53.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5318, 4863
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3388
$N(param)_{refined}$:	335
Program:	SHELXTL [6]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1N)	2*i*		0.1762	0.2969	0.8532	0.072
H(2A)	2*i*		0.5079	–0.2052	0.5468	0.087
H(3A)	2*i*		0.6636	–0.2814	0.6887	0.091
H(5A)	2*i*		0.4092	0.0160	0.8639	0.060
H(11A)	2*i*	0.399	0.3359	0.4521	0.7307	0.072
H(11B)	2*i*	0.601	0.2679	0.4848	0.7167	0.072
H(12A)	2*i*	0.399	0.4749	0.3774	0.7872	0.090
H(12B)	2*i*	0.399	0.3876	0.3865	0.9063	0.090
H(13A)	2*i*	0.399	0.5075	0.5517	0.8692	0.123
H(13B)	2*i*	0.399	0.4227	0.6047	0.7626	0.123
H(14A)	2*i*	0.399	0.3026	0.7340	0.8993	0.113
H(14B)	2*i*	0.399	0.2818	0.6133	0.9775	0.113
H(15A)	2*i*	0.399	0.1298	0.7132	0.7840	0.142
H(15B)	2*i*	0.399	0.0481	0.7219	0.9048	0.142
H(16A)	2*i*	0.399	0.0193	0.5429	0.8180	0.112
H(16B)	2*i*	0.399	0.0937	0.4953	0.9299	0.112
H(12C)	2*i*	0.601	0.2569	0.4421	0.9626	0.096
H(12D)	2*i*	0.601	0.4240	0.3862	0.9054	0.096
H(13C)	2*i*	0.601	0.3849	0.5908	0.9740	0.108
H(13D)	2*i*	0.601	0.4398	0.5926	0.8475	0.108
H(14C)	2*i*	0.601	0.1321	0.6974	0.9380	0.087
H(14D)	2*i*	0.601	0.2365	0.7802	0.8770	0.087
H(15C)	2*i*	0.601	0.2386	0.6975	0.7122	0.098
H(14D)	2*i*	0.601	0.0671	0.7596	0.7594	0.098
H(16C)	2*i*	0.601	0.0456	0.5547	0.8262	0.082
H(16D)	2*i*	0.601	0.0981	0.5550	0.6996	0.082
H(18A)	2*i*		–0.0858	0.0143	0.9218	0.089
H(18B)	2*i*		–0.1461	–0.0458	0.8341	0.089
H(19A)	2*i*		–0.3432	0.1188	0.9075	0.129
H(19B)	2*i*		–0.3061	0.1590	0.7824	0.129
H(19C)	2*i*		–0.2478	0.2170	0.8723	0.129
H(21A)	2*i*		–0.1576	0.5111	0.6069	0.147
H(21B)	2*i*		–0.1424	0.4547	0.4934	0.147
H(22A)	2*i*		–0.3815	0.5091	0.5677	0.305
H(22B)	2*i*		–0.3330	0.4132	0.6724	0.305
H(22C)	2*i*		–0.3185	0.3584	0.5582	0.305
H(23A)	2*i*		0.1617	0.0348	0.4006	0.135
H(23B)	2*i*		0.2225	–0.1092	0.4528	0.135
H(23C)	2*i*		0.3390	–0.0370	0.3941	0.135

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*i*		0.3165(2)	0.2985(1)	0.6119(1)	0.098(1)	0.0635(8)	0.0604(8)	–0.0443(8)	0.0267(7)	–0.0177(6)
O(2)	2*i*		0.0701(2)	0.1572(2)	0.8865(1)	0.0776(9)	0.106(1)	0.0490(8)	–0.0496(8)	0.0113(6)	–0.0166(7)
O(3)	2*i*		–0.0091(1)	0.0611(1)	0.7668(1)	0.0623(8)	0.0723(8)	0.0616(8)	–0.0361(7)	0.0061(6)	–0.0075(6)
O(4)	2*i*		0.0097(3)	0.2437(2)	0.4455(2)	0.150(2)	0.153(2)	0.077(1)	–0.040(2)	–0.050(1)	0.014(1)
O(5)	2*i*		–0.0446(2)	0.3205(2)	0.6106(1)	0.071(1)	0.094(1)	0.083(1)	–0.0056(8)	–0.0128(8)	0.0076(9)
O(6)	2*i*		0.7537(2)	–0.2910(2)	0.8655(2)	0.088(1)	0.128(2)	0.160(2)	0.042(1)	–0.003(1)	0.006(2)
O(7)	2*i*		0.6153(2)	–0.1393(2)	0.9624(2)	0.083(1)	0.095(1)	0.099(1)	–0.0142(9)	–0.024(1)	0.005(1)
N(1)	2*i*		0.2305(2)	0.3230(1)	0.7891(1)	0.084(1)	0.0489(8)	0.0505(8)	–0.0277(8)	0.0085(7)	–0.0121(7)
N(2)	2*i*		0.6461(2)	–0.1932(2)	0.8792(2)	0.048(1)	0.069(1)	0.104(2)	–0.0086(9)	0.002(1)	0.017(1)
C(1)	2*i*		0.3651(2)	–0.0482(2)	0.6204(2)	0.070(1)	0.050(1)	0.055(1)	–0.0342(9)	0.0174(9)	–0.0168(8)
C(2)	2*i*		0.4882(3)	–0.1612(2)	0.6099(2)	0.091(2)	0.052(1)	0.076(1)	–0.034(1)	0.034(1)	–0.029(1)
C(3)	2*i*		0.5801(3)	–0.2065(2)	0.6946(2)	0.071(1)	0.047(1)	0.099(2)	–0.015(1)	0.027(1)	–0.013(1)
C(4)	2*i*		0.5490(2)	–0.1414(2)	0.7879(2)	0.051(1)	0.049(1)	0.081(1)	–0.0164(8)	0.0129(9)	0.0007(9)
C(5)	2*i*		0.4285(2)	–0.0274(2)	0.8005(2)	0.0483(9)	0.0449(9)	0.057(1)	–0.0176(7)	0.0068(8)	–0.0054(7)
C(6)	2*i*		0.3393(2)	0.0180(2)	0.7146(1)	0.0506(9)	0.0409(8)	0.0514(9)	–0.0208(7)	0.0107(7)	–0.0106(7)
C(7)	2*i*		0.2014(2)	0.1399(2)	0.7029(1)	0.0490(9)	0.0471(9)	0.0440(9)	–0.0179(7)	0.0036(7)	–0.0077(7)
C(8)	2*i*		0.1577(2)	0.1310(2)	0.5905(1)	0.065(1)	0.067(1)	0.0457(9)	–0.0344(9)	0.0003(8)	–0.0054(8)
C(9)	2*i*		0.2501(2)	0.0221(2)	0.5454(2)	0.085(1)	0.073(1)	0.0468(9)	–0.054(1)	0.0110(9)	–0.0135(9)
C(10)	2*i*		0.2541(2)	0.2636(2)	0.6985(1)	0.0498(9)	0.0409(8)	0.0496(9)	–0.0117(7)	0.0041(7)	–0.0081(7)
C(11)	2*i*		0.2600(2)	0.4476(2)	0.7916(2)	0.082(1)	0.045(1)	0.053(1)	–0.0214(9)	0.0046(9)	–0.0103(8)
C(12′)	2*i*	0.399(7)	0.3900(8)	0.4308(6)	0.8328(9)	0.063(4)	0.057(3)	0.113(6)	–0.028(3)	–0.018(4)	0.000(4)
C(13′)	2*i*	0.399	0.413(1)	0.564(1)	0.837(1)	0.098(6)	0.090(6)	0.142(8)	–0.057(5)	–0.022(6)	–0.015(6)
C(14′)	2*i*	0.399	0.285(2)	0.650(1)	0.9014(9)	0.15(2)	0.066(9)	0.093(7)	–0.054(8)	–0.020(9)	–0.021(8)
C(15′)	2*i*	0.399	0.131(1)	0.6688(8)	0.858(1)	0.107(6)	0.068(5)	0.18(1)	–0.015(4)	–0.001(7)	–0.064(7)
C(16′)	2*i*	0.399	0.1086(7)	0.5346(7)	0.856(1)	0.060(3)	0.072(4)	0.150(8)	–0.010(3)	0.003(4)	–0.055(5)
C(12)	2*i*	0.601	0.3277(8)	0.4554(4)	0.8996(5)	0.104(4)	0.050(2)	0.093(3)	–0.016(2)	–0.052(3)	–0.007(2)
C(13)	2*i*	0.601	0.356(1)	0.5859(6)	0.9022(8)	0.111(6)	0.052(3)	0.121(5)	–0.023(4)	–0.060(5)	–0.013(4)
C(14)	2*i*	0.601	0.2135(9)	0.6985(6)	0.8792(6)	0.094(5)	0.046(3)	0.082(4)	–0.021(3)	–0.015(3)	–0.015(2)
C(15)	2*i*	0.601	0.1606(7)	0.6890(4)	0.7716(5)	0.111(4)	0.047(2)	0.090(3)	–0.015(2)	–0.040(3)	–0.002(2)
C(16)	2*i*	0.601	0.1300(5)	0.5589(3)	0.7708(5)	0.078(3)	0.054(2)	0.078(3)	–0.017(2)	–0.033(2)	–0.006(2)
C(17)	2*i*		0.0788(2)	0.1246(2)	0.7967(1)	0.0490(9)	0.054(1)	0.048(1)	–0.0184(8)	0.0014(7)	–0.0046(8)
C(18)	2*i*		–0.1249(2)	0.0318(2)	0.8503(2)	0.073(1)	0.087(2)	0.071(1)	–0.048(1)	0.008(1)	0.002(1)
C(19)	2*i*		–0.2679(3)	0.1411(2)	0.8534(2)	0.069(1)	0.102(2)	0.090(2)	–0.036(1)	0.015(1)	–0.020(1)
C(20)	2*i*		0.0337(3)	0.2354(2)	0.5401(2)	0.084(2)	0.092(2)	0.062(1)	–0.045(1)	–0.021(1)	0.014(1)
C(21)	2*i*		–0.1636(3)	0.4375(3)	0.5724(3)	0.085(2)	0.120(2)	0.145(3)	–0.017(2)	–0.034(2)	0.044(2)
C(22)	2*i*		–0.3101(4)	0.4289(4)	0.5943(5)	0.088(2)	0.125(3)	0.345(7)	–0.004(2)	–0.007(3)	0.080(4)
C(23)	2*i*		0.2427(3)	–0.0267(3)	0.4387(2)	0.129(2)	0.113(2)	0.059(1)	–0.081(2)	0.010(1)	–0.029(1)

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