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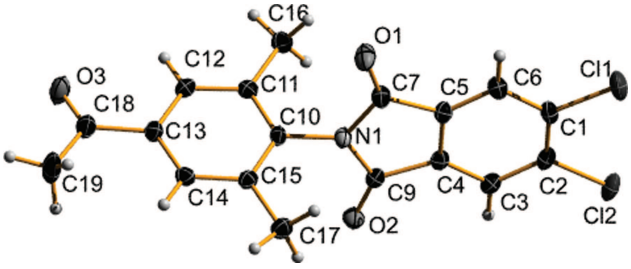
Crystal structure of 2-(4-acetyl-2,6-dimethylphenyl)-5,6-dichloro-1*H*-isoindole-1,3(2*H*)-dione, $C_{18}H_{13}Cl_2NO_3$



DOI 10.1515/ncrs-2015-0021
Received November 19, 2015; accepted January 18, 2016; available online February 25, 2016

Abstract

$C_{18}H_{13}Cl_2NO_3$, monoclinic, $P2_1/n$ (no. 14), $a = 13.6464(2)$ Å, $b = 18.1592(3)$ Å, $c = 13.9829(2)$ Å, $\beta = 107.557(1)°$, $V = 3303.66(9)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0291$, $wR_{ref}(F^2) = 0.0768$, $T = 296(2)$ K.

CCDC no.: 1434982

Only one of two molecules of the title structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

4,5-Dichlorophthalic anhydride (0.54 g, 2.4 mmol) and 4-carbonylmethyl-2,6-dimethylaniline (0.398 g, 2.4 mmol) were mixed in a flask, that was evacuated and filled with nitrogen, then the mixture were dissolved in glacial acetic acid (10 mL) and refluxed for 3 h. The solution was removed under reduced pressure until the volume reached 5 mL. After addition of 10 mL of acetic anhydride, the solution was refluxed again for 20 h. The solvents were evaporated under reduced pressure.

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Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.20×0.20×0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.09 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22286, 6472
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5809
$N(param)_{refined}$:	439
Programs:	Bruker software [2], SHELXL [3, 4]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4e	0.5407	0.6574	0.5974	0.025
H(6)	4e	0.2174	0.6432	0.6504	0.025
H(12)	4e	0.2269	0.3068	0.3138	0.022
H(14)	4e	0.1858	0.4667	0.1070	0.024
H(16A)	4e	0.2832	0.3314	0.4861	0.035
H(16B)	4e	0.2427	0.4065	0.5145	0.035
H(16C)	4e	0.3578	0.3989	0.5166	0.035
H(17A)	4e	0.2052	0.5927	0.1525	0.038
H(17B)	4e	0.3101	0.6015	0.2373	0.038
H(17C)	4e	0.2064	0.6146	0.2613	0.038
H(19A)	4e	0.1012	0.3000	−0.0295	0.064
H(19B)	4e	0.1808	0.3647	−0.0099	0.064
H(19C)	4e	0.0725	0.3771	0.0047	0.064
H(23)	4e	0.7789	0.9323	0.4483	0.025
H(26)	4e	1.1062	0.9056	0.4068	0.025
H(32)	4e	1.0054	0.5335	0.6280	0.021
H(34)	4e	1.0771	0.6561	0.8781	0.026
H(36A)	4e	1.0189	0.6554	0.4627	0.037
H(36B)	4e	0.9039	0.6664	0.4591	0.037
H(36C)	4e	0.9506	0.5869	0.4672	0.037
H(37A)	4e	1.0718	0.7856	0.8734	0.050
H(37B)	4e	0.9817	0.8153	0.7832	0.050
H(37C)	4e	1.0956	0.8214	0.7808	0.050
H(39A)	4e	1.0751	0.4278	0.7442	0.041
H(39B)	4e	0.9597	0.4485	0.7282	0.041
H(39C)	4e	1.0228	0.4080	0.8265	0.041

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.3584(1)	0.69155(8)	0.6986(1)	0.0271(7)	0.0169(7)	0.0153(6)	0.0046(6)	0.0033(6)	−0.0011(5)
C(2)	4*e*	0.4554(1)	0.69454(8)	0.6847(1)	0.0233(7)	0.0148(7)	0.0192(7)	−0.0011(5)	−0.0015(6)	−0.0006(5)
C(3)	4*e*	0.4765(1)	0.65522(8)	0.6077(1)	0.0188(7)	0.0188(7)	0.0233(7)	−0.0010(5)	0.0049(6)	−0.0001(6)
C(4)	4*e*	0.3982(1)	0.61298(7)	0.5474(1)	0.0190(7)	0.0161(7)	0.0185(6)	0.0015(5)	0.0045(5)	0.0003(5)
C(5)	4*e*	0.3037(1)	0.60775(8)	0.5643(1)	0.0177(7)	0.0191(7)	0.0188(7)	0.0006(5)	0.0025(5)	−0.0008(5)
C(6)	4*e*	0.2812(1)	0.64672(8)	0.6396(1)	0.0187(7)	0.0227(7)	0.0190(7)	0.0036(6)	0.0039(5)	−0.0018(6)
C(7)	4*e*	0.2376(1)	0.55690(8)	0.4889(1)	0.0180(7)	0.0232(7)	0.0215(7)	0.0008(6)	0.0046(6)	−0.0049(6)
C(9)	4*e*	0.3952(1)	0.56728(8)	0.4580(1)	0.0187(7)	0.0167(7)	0.0201(7)	−0.0014(5)	0.0044(6)	−0.0007(5)
C(10)	4*e*	0.2638(1)	0.48193(8)	0.3483(1)	0.0146(6)	0.0192(7)	0.0186(6)	−0.0017(5)	0.0050(5)	−0.0055(5)
C(11)	4*e*	0.2597(1)	0.40774(8)	0.3747(1)	0.0139(6)	0.0221(7)	0.0179(6)	0.0003(5)	0.0042(5)	0.0004(5)
C(12)	4*e*	0.2293(1)	0.35645(8)	0.2981(1)	0.0172(7)	0.0158(7)	0.0226(7)	−0.0005(5)	0.0052(5)	0.0010(5)
C(13)	4*e*	0.2022(1)	0.37810(8)	0.1978(1)	0.0183(7)	0.0194(7)	0.0197(7)	−0.0013(5)	0.0043(5)	−0.0020(5)
C(14)	4*e*	0.2051(1)	0.45238(8)	0.1740(1)	0.0208(7)	0.0216(7)	0.0160(6)	−0.0008(6)	0.0048(5)	0.0019(5)
C(15)	4*e*	0.2367(1)	0.50572(8)	0.2490(1)	0.0167(7)	0.0182(7)	0.0212(7)	−0.0005(5)	0.0063(5)	−0.0003(5)
C(16)	4*e*	0.2885(1)	0.38398(9)	0.4827(1)	0.0235(7)	0.0266(8)	0.0191(7)	−0.0012(6)	0.0043(6)	0.0017(6)
C(17)	4*e*	0.2399(1)	0.58589(8)	0.2227(1)	0.0291(8)	0.0192(7)	0.0278(8)	−0.0023(6)	0.0083(6)	0.0008(6)
C(18)	4*e*	0.1692(1)	0.31915(9)	0.1190(1)	0.0298(8)	0.0230(8)	0.0234(7)	−0.0038(6)	0.0052(6)	−0.0033(6)
C(19)	4*e*	0.1271(2)	0.3423(1)	0.0114(1)	0.069(1)	0.0276(9)	0.0213(8)	−0.0084(9)	−0.0005(8)	−0.0055(7)
C(21)	4*e*	0.9722(1)	0.96336(8)	0.3604(1)	0.0256(7)	0.0157(7)	0.0197(7)	−0.0034(6)	0.0077(6)	0.0036(5)
C(22)	4*e*	0.8759(1)	0.97215(8)	0.3743(1)	0.0236(7)	0.0163(7)	0.0230(7)	0.0016(6)	0.0036(6)	0.0054(6)
C(23)	4*e*	0.8435(1)	0.92703(8)	0.4396(1)	0.0202(7)	0.0179(7)	0.0261(7)	0.0011(6)	0.0078(6)	0.0029(6)
C(24)	4*e*	0.9120(1)	0.87428(7)	0.4907(1)	0.0211(7)	0.0135(6)	0.0192(6)	−0.0009(5)	0.0062(5)	0.0003(5)
C(25)	4*e*	1.0087(1)	0.86713(7)	0.4792(1)	0.0201(7)	0.0147(7)	0.0214(7)	−0.0003(5)	0.0048(6)	0.0007(5)
C(26)	4*e*	1.0411(1)	0.91092(8)	0.4143(1)	0.0199(7)	0.0194(7)	0.0248(7)	−0.0006(6)	0.0081(6)	0.0024(6)
C(27)	4*e*	1.0621(1)	0.80419(8)	0.5419(1)	0.0216(7)	0.0173(7)	0.0253(7)	−0.0005(6)	0.0063(6)	0.0034(6)
C(29)	4*e*	0.8976(1)	0.81439(8)	0.5581(1)	0.0227(7)	0.0146(7)	0.0204(7)	0.0015(5)	0.0070(6)	0.0009(5)
C(30)	4*e*	1.0094(1)	0.71054(7)	0.6479(1)	0.0179(7)	0.0150(7)	0.0234(7)	0.0015(5)	0.0057(6)	0.0062(5)
C(31)	4*e*	0.9970(1)	0.64272(8)	0.5993(1)	0.0147(6)	0.0195(7)	0.0192(7)	0.0015(5)	0.0047(5)	0.0021(5)
C(32)	4*e*	1.0139(1)	0.57950(8)	0.6586(1)	0.0160(6)	0.0147(6)	0.0217(7)	0.0003(5)	0.0042(5)	0.0001(5)
C(33)	4*e*	1.0434(1)	0.58422(8)	0.7626(1)	0.0149(6)	0.0173(7)	0.0209(7)	−0.0001(5)	0.0030(5)	0.0030(5)
C(34)	4*e*	1.0562(1)	0.65312(8)	0.8085(1)	0.0229(7)	0.0219(7)	0.0170(7)	−0.0015(6)	0.0014(6)	0.0009(6)
C(35)	4*e*	1.0383(1)	0.71752(8)	0.7521(1)	0.0232(7)	0.0170(7)	0.0240(7)	−0.0015(6)	0.0038(6)	0.0005(6)
C(36)	4*e*	0.9647(1)	0.63734(8)	0.4869(1)	0.0310(8)	0.0226(7)	0.0203(7)	0.0035(6)	0.0084(6)	0.0033(6)
C(37)	4*e*	1.0477(2)	0.79166(9)	0.8019(1)	0.050(1)	0.0184(8)	0.0271(8)	−0.0027(7)	0.0038(7)	−0.0023(6)
C(38)	4*e*	1.0590(1)	0.51677(8)	0.8268(1)	0.0185(7)	0.0199(7)	0.0217(7)	−0.0002(6)	0.0029(6)	0.0036(6)
C(39)	4*e*	1.0262(1)	0.44366(8)	0.7769(1)	0.0319(8)	0.0174(7)	0.0268(8)	−0.0035(6)	−0.0004(6)	0.0056(6)
N(1)	4*e*	0.29703(9)	0.53502(7)	0.42776(9)	0.0176(6)	0.0208(6)	0.0199(6)	−0.0027(5)	0.0051(5)	−0.0059(5)
N(2)	4*e*	0.99150(9)	0.77612(6)	0.58751(9)	0.0219(6)	0.0156(6)	0.0234(6)	0.0020(5)	0.0076(5)	0.0057(5)
O(1)	4*e*	0.15112(8)	0.53703(7)	0.47978(8)	0.0174(5)	0.0399(7)	0.0345(6)	−0.0058(5)	0.0089(5)	−0.0159(5)
O(2)	4*e*	0.46034(8)	0.55874(6)	0.41719(8)	0.0222(5)	0.0313(6)	0.0280(5)	−0.0058(4)	0.0123(4)	−0.0082(5)
O(3)	4*e*	0.1762(1)	0.25459(6)	0.14199(9)	0.0557(8)	0.0183(6)	0.0297(6)	−0.0045(5)	0.0058(6)	−0.0038(5)
O(4)	4*e*	1.14673(8)	0.78024(6)	0.55197(9)	0.0224(6)	0.0291(6)	0.0476(7)	0.0070(5)	0.0128(5)	0.0152(5)
O(5)	4*e*	0.82262(8)	0.79891(6)	0.58173(8)	0.0280(6)	0.0227(5)	0.0364(6)	0.0037(4)	0.0181(5)	0.0088(5)
O(6)	4*e*	1.09765(9)	0.52141(6)	0.91730(8)	0.0373(6)	0.0231(5)	0.0195(5)	−0.0011(5)	0.0022(5)	0.0056(4)
Cl(1)	4*e*	0.33039(3)	0.74560(2)	0.78865(3)	0.0363(2)	0.0295(2)	0.0213(2)	0.0041(2)	0.0055(2)	−0.0099(1)
Cl(2)	4*e*	0.55214(3)	0.74776(2)	0.76193(3)	0.0295(2)	0.0247(2)	0.0298(2)	−0.0075(2)	−0.0001(2)	−0.0092(2)
Cl(3)	4*e*	1.00666(3)	1.01528(2)	0.27283(3)	0.0335(2)	0.0279(2)	0.0304(2)	0.0003(2)	0.0145(2)	0.0135(2)
Cl(4)	4*e*	0.79385(3)	1.03979(2)	0.30874(3)	0.0287(2)	0.0332(2)	0.0493(2)	0.0102(2)	0.0113(2)	0.0259(2)

The residue was neutralized with a solution of sodium bicarbonate (4%) and stirred for 30 min. The organic phase was extracted with CH_2Cl_2 and washed with H_2O (3 * 20 mL), dried (Na_2SO_4), and the solvent was evaporated under reduced pressure. The products were separated by column chromatography (cyclohexane/EtoAc 10:1), yielded the product (0.71 g, 81.98%) as a white solid. Single crystals were grown by slow evaporation from a 3/1 (v/v) solution of $CHCl_3$ and EtOAc.

Experimental details

All the hydrogen atoms were generated geometrically.

Discussion

The title compound, $C_{18}H_{13}Cl_2NO_3$, is a product of amidolysis reaction of 4,5-dichlorophthalic anhydride and 4-carbonylmethyl-2,6-dimethylaniline. The asymmetric unit of this structure contains two discrete molecules. In the two independent molecules, the dihedral angles between the substituted phenyl rings and benzofuranyl moieties are 80.00(4) and 76.34(5)°, respectively.

Among various classes of receptor molecules, the quinoxaline-bridged resorcin arene are particularly fascinating because of their ability to undergo a triggered conformational transition from a contracted vase state to an expanded kite state [1]. The compound we synthetized can be used to bridge different arm molecules with the dicavitand.

Acknowledgements: This project was supported by National Natural Science Foundation of China (No. 2100212127 and No. 21255110517), and Key Laboratory of Resources Chemistry of Nonferrous Metals, Ministry of Education (Central South University). The authors thank the responsible editor for supplying the figure.

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