Crystal Structure of Dimethyl 2-[(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)oxy]-2-butenedioate

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The crystal structure of dimethyl 2-[(1,3-dioxo-1,3-dihydro-2*H*-isoindol-2-yl)oxy]-2-butenedioate is monoclinic, space group $P2_1/c$ with $a = 7.9785(16)$, $b = 24.801(6)$, $c = 7.9550(16)$ Å, $\beta = 118.904(13)^\circ$ and $Z = 4$. The crystal structure was solved by direct methods and refined by full-matrix least squares to final values of $R1 = 0.0567$ and $wR2 = 0.0884$ with 1927 reflections ($I > 2\sigma(I)$).

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Isoindoles and their derivatives are very useful as starting materials to produce pharmaceutically and industrially important compounds.^{1,2} Some of these compounds are selective monoamine oxidase inhibitors, and therefore are useful for treating or preventing diseases mediated by monoamine oxidase.¹ In addition to being used in the pharmaceutical industry, isoindoles also find a wide variety of application in the dye and polymer industries.³ In this paper, we show the structure of dimethyl 2-[(1,3-dioxo-1,3-dihydro-2*H*-isoindol-2-yl)oxy]-2-butenedioate.

The compound was prepared by the following method. To a magnetically stirred solution of *N*-hydroxyphthalimide (0.163 g, 1 mmol) in acetone-water (5:1) (6 ml) was added dropwise a mixture of dimethyl acetylenedicarboxylate (0.14 ml, 1 mmol) in acetone (1 ml) at -10°C over 15 min. The mixture was then stirred at -10°C for 30 min. The mixture was allowed to warm up to room temperature and was then stirred for 6 h. The mixture was cloudy white, and it was filtered, the filtrate being evaporated to dryness as a white solid. Yield: 0.28 g (91%); m.p: 128.0–130°C. Found: C, 55.21; H, 3.58; N, 4.64; calculated for $\text{C}_{14}\text{H}_{11}\text{NO}_7$; C, 55.09; H, 3.63; N, 4.59%. Single crystals of dimethyl 2-[(1,3-dioxo-1,3-dihydro-2*H*-isoindol-2-yl)oxy]-2-butenedioate were prepared by using a branched tube method from a mixture of ethyl acetate-light petroleum ether (1:10) at 40°C during six weeks. Colorless single crystals of dimethyl 2-[(1,3-dioxo-1,3-dihydro-2*H*-isoindol-2-yl)oxy]-2-butenedioate were collected *via* filtration, washed with a cold

mixture of ethyl acetate-light petroleum ether (1:10) and dried at room temperature.

X-ray measurements were made at 293(2)K using a Siemens R3m/V diffractometer. The intensity data were collected within the range $2.92^\circ \leq \theta \leq 29.062^\circ$ using graphite-monochromated $\text{Mo-K}\alpha$ radiation ($\lambda = 0.71073$ Å). Accurate unit-cell parameters and an orientation matrix for data collection were obtained from the least-squares refinement. The intensities of 3896 reflections were measured, 3614 of which were unique. The structure was solved by direct methods and refined by full-matrix least-squares techniques on F^2 .

Table 1 Crystal data and structure refinement for dimethyl 2-[(1,3-dioxo-1,3-dihydro-2*H*-isoindol-2-yl)oxy]-2-butenedioate

Empirical formula	$\text{C}_{14}\text{H}_{11}\text{NO}_7$
Formula weight	305.24
Temperature	293(2)K
Wavelength	0.71073 Å
Crystal system, space group	monoclinic, $P2_1/c$
Unit cell dimensions	$a = 7.9785(16)$ Å $b = 24.801(6)$ Å $\beta = 118.904(13)^\circ$ $c = 7.9550(16)$ Å
Volume	$1378.0(5)$ Å ³
Z , Calculated density	4, 1.471 Mg/m ³
Absorption coefficient ($\text{Mo K}\alpha$)	0.12 mm ⁻¹
$F(0\ 0\ 0)$	632
Crystal size	$0.45 \times 0.30 \times 0.20$ mm
θ range for data collection	2.92 to 29.06°
Limiting indices	$-9 \leq h \leq 10$, $-33 \leq k \leq 0$, $-10 \leq l \leq 1$
Reflections collected/unique	3896/3614 [$R(\text{int}) = 0.0207$]
Completeness to $\theta = 29.06$	98.3%
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	3614/0/199
Goodness-of-fit on F^2	1.000
Final R indices [for 1927 rfls with $I > 2\sigma(I)$]	$R1 = 0.0567$, $wR2 = 0.0884$
R indices (all data)	$R1 = 0.1545$, $wR2 = 0.1075$
Largest diff. peak and hole (Δ/δ) _{max}	0.177 and -0.147 e.Å ⁻³
	0.000

Fig. 1 Chemical structure of dimethyl 2-[(1,3-dioxo-1,3-dihydro-2*H*-isoindol-2-yl)oxy]-2-butenedioate.

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Fig. 2 (a) ORTEP diagram of dimethyl 2-[(1,3-dioxo-1,3-dihydro-2H-isoindol-2-yl)oxy]-2-butenedioate, Ellipsoids 30% probability. (b) Unit-cell plot of dimethyl 2-[(1,3-dioxo-1,3-dihydro-2H-isoindol-2-yl)oxy]-2-butenedioate.

The H(C) atom positions were calculated and refined in an isotropic approximation in the riding model with the Uiso(H) parameters equal to 1.2 Ueq(Ci), for methyl groups equal to 1.5 Ueq(Cii), where U(Ci) and U(Cii) are, respectively, the equivalent thermal parameters of the carbon atoms to which corresponding H atoms are bonded. All of the calculations were carried out with a PDP-11/23+ computer using the SDP-PLUS program package.⁴ The crystal (Fig. 2a) consists of a discrete molecule. The torsion angles of C9–O3–N1–C8 is 127.07(19)° and the isoindol with a butenedioate group are not coplanar. The bond lengths and angles are within the expected ranges. A search was generally made for C–H...O=C approaches on the packing of this compound. The interesting feature of the compound in the solid state is that there are C–H...O=C interactions, weak hydrogen bonding between the hydrogen atoms of aromatic rings and oxygen atoms. In this compound the C–H...O distances range is 2.56 to 2.65 Å. These values suggest strong interactions within this class of weak noncovalent contacts.⁵

Supplementary Material

Complete bond lengths and angles, coordinates and

Table 2 Atomic coordinates ($\times 10^5$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^5$) for dimethyl 2-[(1,3-dioxo-1,3-dihydro-2H-isoindol-2-yl)oxy]-2-butenedioate. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	y	z	$U(\text{eq})$
O1	1184(2)	15127(6)	6182(2)	535(4)
O2	6409(2)	6317(7)	6636(2)	625(5)
O3	4602(2)	16437(6)	5945(2)	512(4)
O4	2416(2)	11011(6)	2409(2)	658(5)
O5	649(2)	18428(6)	1262(2)	544(4)
O6	4776(3)	32928(7)	5264(3)	913(7)
O7	6222(2)	25942(6)	7118(2)	605(5)
N1	3887(2)	11682(6)	6257(3)	454(4)
C1	2259(3)	11412(8)	6470(3)	382(4)
C2	2209(3)	5730(7)	7021(3)	384(5)
C3	893(3)	3173(9)	7375(3)	513(6)
C4	1200(4)	-2231(9)	7875(4)	620(7)
C5	2769(4)	-4921(9)	7989(4)	626(7)
C6	4087(3)	-2364(8)	7604(3)	518(6)
C7	3783(3)	3057(8)	7125(3)	389(5)
C8	4932(3)	6877(8)	6658(3)	414(5)
C9	3377(3)	19062(8)	4254(3)	395(5)
C10	2099(3)	15630(8)	2557(3)	411(5)
C11	-0592(4)	15585(10)	-523(3)	677(7)
C12	3542(3)	24345(8)	4102(3)	429(5)
C13	4886(3)	28185(8)	5527(3)	464(5)
C14	7609(3)	29584(10)	8537(4)	674(7)

displacement parameters have been deposited at Cambridge Crystallography Data Centre. Supplementary data are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request, by quoting the deposition number (618726) for dimethyl 2-[(1,3-dioxo-1,3-dihydro-2H-isoindol-2-yl)oxy]-2-butenedioate.

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