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Ethyl *N*-(2-butyl-1,3-dioxo-2,3-dihydro-1*H*-isoindol-5-yl)carbamate

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Key indicators

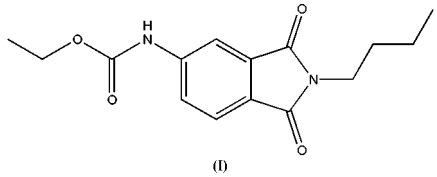
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.062
wR factor = 0.168
Data-to-parameter ratio = 12.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{15}H_{18}N_2O_4$, was synthesized by the triphosgenation of ethanol and *N*-butyl-4-aminophthalimide. There are two independent molecules in the asymmetric unit, which are linked into chains by N—H···O interactions.

Comment

Organic carbamates are valuable synthetic intermediates, and are found in a variety of biologically active compounds (Vauthey *et al.*, 2000). Functionalization of the carbamate moiety offers an attractive method for the generation of derivatives, which may have interesting medicinal and biological properties (Folkmann & Lund, 1990). We are interested in phthalimide derivatives and a search of the literature revealed that some phthalimide derivatives have cytotoxicity (Hall *et al.*, 1995) and anti-HIV activity (Van Derpoorten *et al.*, 1997). It has been assumed that compounds having both phthalimide and carbamate residues in the same molecule may possess some attractive biological activities. In this connection, the structure of the title compound, ethyl *N*-(2-butyl-1,3-dioxo-2,3-dihydro-1*H*-isoindol-5-yl)carbamate, (I), is reported.



(I)

There are two independent molecules of (I) in the asymmetric unit and molecules are connected into chains *via* N—H···O contacts: H···O 2.15 Å, N···O 2.985 (16) Å and N—H···O 163° for N2—H···O6; H···O 2.11 Å, N···O 2.947 (16) Å and N—H···O 163° for N4—H···O2^{i} [symmetry code: (i) *x*, −1 + *y*, *z*] (Fig. 1 and Table 1). This arrangement is similar to that found in phthalimide (Zakaria *et al.*, 2002). The phthalimide group is planar, the mean deviation from the least-squares plane being 0.005 (3) Å [0.015 (4) Å for the second independent molecule]. This observation is different from that in the related compound, *N*-(3-iodopropyl)phthalimide, which is not exactly planar (Chandramohan *et al.*, 2000). The carbamate moiety is effectively planar; the mean deviation of the atoms from this plane is 0.002 (3) Å [0.001 (3) Å], in good agreement with the corresponding results for ethyl *N*-(3-oxobutanoyl)carbamate (Golden *et al.*, 2002). The dihedral angle between the mean planes of the phthalimide and carbamate moieties is 5.7 (3) and 5.9 (3)°, respectively. The *n*-butyl moieties (C1/C2/C3/C4 and C16/C17/C18/C19) and phthalimide groups are folded towards each other,

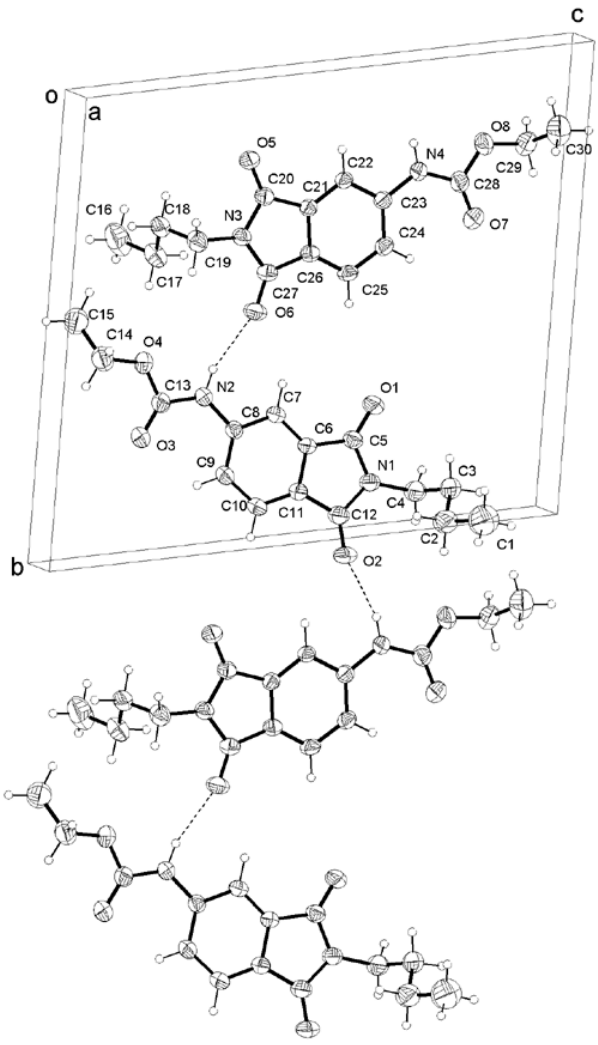


Figure 1
Structure of (I), showing H-bonding interactions and the atomic numbering scheme.

making a dihedral angle of 106.4 (2)° [101.7 (2)°], which is somewhat different from the corresponding angle in the *N*-(3-iodopropyl)phthalimide structure, *viz*. 76.6 (2)° (Chandramohan *et al.*, 2000).

Experimental

N-Butyl-4-aminophthalimide (12 mmol) and triethylamine (12 mmol) in dry chloroform (30 ml) were placed in a 50 ml flask equipped with an N_2 inlet adaptor and a rubber septum, and the mixture was cooled to 263 K with stirring. Triphosgene (4 mmol) in chloroform was added dropwise and the resulting mixture was stirred at 273 K for 3 h and at 293 K for an additional 2 h. The solvent was then evaporated *in vacuo* to give (2-butyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-5-yl)carbamic chloride, (II). (II) was dissolved in dry ethanol (20 ml) with stirring at room temperature for 2 h. After evaporation of the solvent, the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 10:1) to give the title compound, (I). M.p. 413–415 K; IR (KBr): 3340 (N—H), 3080, 2951 (C—H), 1730, 1697 (C═O) cm^{-1}; ^1H NMR ($CDCl_3$, p.p.m.): 0.92 (3H, *t*), 1.27–1.43 (5H, *m*), 1.62 (2H, *s*), 3.63 (2H, *t*), 4.24 (2H, *m*), 6.98 (1H, *s*), 7.72 (2H, *m*), 7.85 (1H, *s*). (I) (50 mg) was dissolved in ethyl acetate (15 ml) and the solution was kept at room temperature for 4 d, yielding colorless single crystals.

Crystal data

$C_{15}H_{18}N_2O_4$	$Z = 4$
$M_r = 290.31$	$D_x = 1.275$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.933$ (2) Å	Cell parameters from 936 reflections
$b = 14.210$ (4) Å	$\theta = 2.8$–$23.1°$
$c = 15.913$ (5) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 99.657$ (6)°	$T = 293$ (2) K
$\beta = 101.373$ (6)°	Plate, colorless
$\gamma = 91.120$ (6)°	0.30 × 0.20 × 0.08 mm
$V = 1512.8$ (8) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	5277 independent reflections
φ and ω scans	2549 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.035$
$T_{min} = 0.808$, $T_{max} = 1.000$	$\theta_{max} = 25.1°$
7713 measured reflections	$h = -7 \rightarrow 8$
	$k = -11 \rightarrow 16$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.062$	$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.168$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.30$ e Å^{-3}
5277 reflections	$\Delta\rho_{min} = -0.31$ e Å^{-3}
418 parameters	

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.220 (4)	N1—C5	1.386 (4)
O4—C13	1.346 (4)	N2—C13	1.350 (4)
O5—C20	1.211 (4)	N3—C27	1.392 (4)
O8—C28	1.349 (4)	N4—C28	1.352 (4)
C13—O4—C14	122.0 (10)	C27—N3—C20	111.5 (3)
C28—O8—C29	120.5 (6)	C28—N4—C23	128.0 (3)
C5—N1—C12	111.4 (3)	O1—C5—C6	129.1 (3)
C13—N2—C8	127.7 (3)	O5—C20—C21	129.1 (3)

All H atoms were included in the riding-model approximation with $U_{iso} = 1.2U_{eq}$ for CH_2 and $1.5U_{eq}$ for CH_3. The terminal ethyl groups in the carbamate residues were found to be disordered. There are two components of the disorder, each with unequal partial occupancies, *viz*. 0.77 (2) and 0.23 (2) in the first molecule, and 0.53 (2) and 0.47 (2) in the second. The distances C14—C15, C14′—C15′, C29—C30 and C29′—C30′ were restrained to 1.54 Å and C14—O4, C14′—O4′, C29—O8 and C29′—O8′ were restrained to 1.45 Å.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1998). *SMART, SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chandramohan, K., Ravikumar, K. & Rajendar, S. (2000). *Acta Cryst.* C**56**, e298–e299.

Folkmann, M. & Lund, F. J. (1990). *Synthesis*, pp. 1159–1166.
Golden, M. C., McGill, R. J. & Heimer, N. E. (2002). *Acta Cryst.* E**58**, o553–o554.
Hall, I. H., Wong, O. T. & Scovill, J. P. (1995). *Biomed. Pharmacother.* **49**, 251–258.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Van Derpoorten, K., Balzarini, J., De Clercq, E. & Poupaert, J. H. (1997). *Biomed. Pharmacother.* **51**, 464–468.
Vauthey, I., Valot, F., Gozzi, C., Fache, F. & Lemaire, M. (2000). *Tetrahedron Lett.* **33**, 6347–6350.
Zakaria, C. M., Low, J. N. & Glidewell, C. (2002). *Acta Cryst.* C**58**, o9–o10.