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Methyl (*E*)-3-Benzamido-2-bromoacrylate

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Abstract. $C_{11}H_{10}BrNO_3$, $M_r = 284{\cdot}1$, orthorhombic, $Pna2_1$, $a = 9{\cdot}915$ (1), $b = 17{\cdot}834$ (1), $c = 6{\cdot}442$ (1) Å, $V = 1139$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}657$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 3{\cdot}501$ mm^{-1}, $F(000) = 568$, $T = 295$ (2) K, $R = 0{\cdot}033$ for 1151 observed reflections. The structure investigation determines the stereochemistry of the title compound as *E*. Bond distances and angles are as expected. The non-phenyl portion of the molecule is planar [maximum deviation: 0·106 (5) Å] and forms a dihedral angle of 15·4° with the phenyl ring. There are no significant intermolecular contacts in the crystal lattice.

Experimental. A mixture of *N*-benzoyl-β-alanine methyl ester (0·3 g, 1·4 mmol), *N*-bromosuccinimide (0·26 g, 1·4 mmol) and 2,2′-azobisisobutyronitrile (*ca* 5 mg) in CCl_4 (15 ml) was heated for 2 h at reflux under N_2 whilst being irradiated with a 250 W Hg lamp. The filtrate was concentrated *in vacuo* and chromatographed on silica to give methyl (*E*)-3-benzamido-2-bromoacrylate (132 mg, 32%), isomer *A*, m.p. 371–372 K, found: C, 46·48; H, 3·53%; $C_{11}H_{10}BrNO_3$ requires C, 46·50; H, 3·54% and methyl (*Z*)-3-benzamido-2-bromoacrylate (62 mg, 15%), isomer *B*, m.p. 395·5–396·5 K, found: C, 46·51; H, 3·67%; $C_{11}H_{10}BrNO_3$ requires C, 46·50; H, 3·54%. Crystals of isomer *A* grown by slow evaporation of hexane into an ethyl acetate solution of the compound. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ-scan technique. Cell parameters on crystal 0·18 × 0·12 × 0·75 mm by least squares on 25 reflections ($8 \le \theta \le 12°$) (de Boer & Duisenberg, 1984). Analytical absorption correction applied; max. and min. transmission factors 0·710 and 0·520 (Sheldrick, 1976). Total of 2588 reflections ($1{\cdot}5 \le \theta \le 27{\cdot}5°$) measured in the range $0 \le h \le 12$, $-23 \le k \le 12$, $0 \le l \le 8$. No significant variation in the net intensities of three reference reflections ($2\bar{2}5$, $2\bar{3}4$, $1\bar{4}2$) measured every 7200 s. 1486 unique reflections (R_{int} 0·024) and 1151 satisfied $I \ge 2{\cdot}5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement of 173 parameters based on *F* (Sheldrick, 1976). Anisotropic

Table 1. *Fractional atomic coordinates and* B_{eq} *values* (Å^2)

$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3$.

	x	*y*	*z*	B_{eq}
Br(2)	−0·06102 (4)	−0·05594 (3)	0	4·77
O(1)	−0·2244 (4)	0·0015 (3)	−0·3736 (8)	6·49
O(1′)	−0·3878 (5)	−0·0834 (3)	−0·3656 (7)	4·87
O(4)	−0·4316 (3)	−0·2594 (2)	0·1777 (6)	4·54
N(3)	−0·2455 (4)	−0·1872 (3)	0·1491 (7)	3·54
C(1)	−0·2761 (5)	−0·0516 (3)	−0·2937 (9)	3·54
C(1′)	−0·4478 (6)	−0·0519 (4)	−0·5501 (9)	5·41
C(2)	−0·2261 (5)	−0·0903 (3)	−0·1082 (8)	3·14
C(3)	−0·2894 (5)	−0·1479 (3)	−0·0213 (10)	3·15
C(4)	−0·3211 (5)	−0·2422 (3)	0·2438 (8)	3·27
C(41)	−0·2616 (5)	−0·2767 (3)	0·4353 (7)	3·11
C(42)	−0·1609 (5)	−0·2417 (3)	0·5468 (8)	3·55
C(43)	−0·1157 (6)	−0·2729 (4)	0·7321 (10)	4·62
C(44)	−0·1690 (6)	−0·3382 (4)	0·7993 (10)	5·01
C(45)	−0·2665 (7)	−0·3750 (4)	0·6880 (11)	5·21
C(46)	−0·3127 (5)	−0·3445 (3)	0·5021 (15)	4·44

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Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

C(1)—O(1)	1·195 (6)	C(1)—O(1′)	1·327 (6)
O(1′)—C(1′)	1·443 (7)	C(1)—C(2)	1·466 (7)
C(2)—Br(2)	1·882 (5)	C(2)—C(3)	1·328 (7)
C(3)—N(3)	1·372 (7)	N(3)—C(4)	1·377 (7)
C(4)—O(4)	1·214 (5)	C(4)—C(41)	1·499 (7)
C(1)—O(1′)—C(1′)	117·7 (5)	O(1)—C(1)—O(1′)	123·1 (5)
O(1)—C(1)—C(2)	125·4 (5)	O(1′)—C(1)—C(2)	111·5 (5)
C(1)—C(2)—Br(2)	116·3 (4)	C(1)—C(2)—C(3)	123·2 (5)
Br(2)—C(2)—C(3)	120·4 (4)	C(2)—C(3)—N(3)	125·6 (5)
C(3)—N(3)—C(4)	123·0 (4)	N(3)—C(4)—O(4)	121·0 (5)
N(3)—C(4)—C(41)	116·2 (4)	O(4)—C(4)—C(41)	122·7 (5)

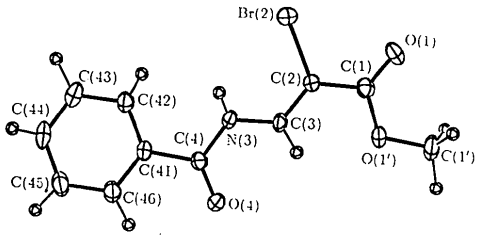

Fig. 1. Molecular structure and numbering scheme for $C_{11}H_{10}BrNO_3$. H atoms are labelled according to the atom to which they are bonded (Johnson, 1971).

thermal parameters for non-H atoms and isotropic thermal parameters for H atoms which were located from a difference map except for methyl H atoms which were included at their calculated positions (C—H 0·97 Å) and refined with a common isotropic thermal parameter. At convergence $R = 0{\cdot}033$, $wR = 0{\cdot}033$, $w = 1{\cdot}89/[\sigma^2(F) + 0{\cdot}0003F^2]$, $S = 1{\cdot}97$, $(\Delta/\sigma)_{max} \leq 0{\cdot}001$, $\Delta\rho_{max} = 0{\cdot}41$, $\Delta\rho_{min} = -0{\cdot}46$ e Å^{-3}; no extinction correction. Scattering factors for H, C, N and O given in *SHELX*76 (Sheldrick, 1976) and those for neutral Br corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974). All calculations on a SUN4/280 computer system. Atomic parameters are given in Table 1, selected parameters in Table 2* and the numbering scheme used is shown in Fig. 1, drawn with *ORTEP* (Johnson, 1971) at 25% probability ellipsoids.

Related literature. Owing to similarities in the spectroscopic data for isomers *A* and *B* it was necessary to characterize one of these by X-ray methods to determine the stereochemistry as *E* or *Z*. The structure determination of the title compound forms part of a wider study of the synthesis of halogenated amino acid derivatives (Burgess, Easton & Hay, 1989).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, all bond distances and angles, and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52564 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of 2,5,7,9-Tetranitro-8-oxo-2,5,7,9-tetraazabicyclo[4.3.0]nonane (I) and 2,5,7,9-Tetranitro-8-acetoxy-2,5,7,9-tetraazabicyclo[4.3.0]nonane (II)

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Abstract. (I): Perhydro-1,3,5,7-tetranitroimidazo[4,5-*b*]pyrazin-2-one, $C_5H_6N_8O_9$, $M_r = 322{\cdot}15$, trigonal, $P3_1$ (arbitrarily chosen over its enantiomorphic counterpart $P3_2$), $a = 10{\cdot}923$ (4), $c = 7{\cdot}888$ (4) Å, $V = 815{\cdot}7$ (2) Å^3, $Z = 3$, $D_x = 1{\cdot}969$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}18$ mm^{-1}, $F(000) = 492$, $T = 295$ K, final $R = 0{\cdot}056$, $wR = 0{\cdot}054$ for 895 observed reflections. (II): Perhydro-1,3,5,7-tetranitroimidazo[4,5-*b*]pyrazin-2-yl acetate, $C_7H_{10}N_8O_{10}$, $M_r = 366{\cdot}21$, monoclinic, $P2_1/a$, $a = 11{\cdot}206$ (2), $b =$

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