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3-[(4-Bromo-1-methyl-1*H*-pyrrol-2-ylcarbonyl)-amino]propanoic acid

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.036
wR factor = 0.100
Data-to-parameter ratio = 12.5

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

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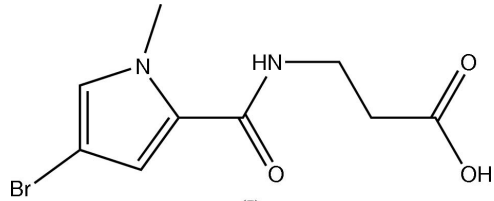
The title compound, $C_9H_{11}BrN_2O_3$, was synthesized by condensation of β-alanine methyl ester with 4-bromo-1-methyl-2-(trichloroacetyl)pyrrole at room temperature. In the crystal structure, intermolecular N—H···O and O—H···O hydrogen-bond interactions link the molecules into extended ribbons parallel to the *a* axis.

Comment

Pyrrole derivatives are well known in many marine organisms (Faulkner, 2001), and some are bioactive substances (Tasdemir *et al.*, 2002). In our search for bioactive compounds, a series of brominated (pyrrol-2-ylcarbonyl)amino acids and their methyl esters, including the title compound, (I), have been synthesized by reaction of β-alanine methyl ester with brominated 2-(trichloroacetyl)pyrrole or brominated 1-methyl-2-(trichloroacetyl)pyrrole, followed by saponification and acidification. Pharmacological studies have shown that (I) moderately inhibits *Streptococcus faecalis* and *Micrococcus luteus*. We report the crystal structure of (I).



(I)

Bond lengths and angles are unexceptional and are in good agreement with the corresponding values in 3-(4-bromo-1*H*-pyrrole-2-carboxamido)propanoic acid (Zeng *et al.*, 2005).

In the crystal structure, the molecules are linked through intermolecular N—H···O hydrogen bonds to give dimeric centrosymmetric $R_2^2(12)$ rings (Table 1). The dimers are connected by strong O—H···O hydrogen-bond interactions, generating ribbons running parallel to the *a* axis (Fig. 2).

Experimental

The hydrochloric acid salt of β-alanine methyl ester (0.70 g, 5 mmol) and 4-bromo-1-methyl-2-(trichloroacetyl)pyrrole (1.53 g, 5 mmol) were added to acetonitrile (12 ml), followed by the dropwise addition of triethylamine (1.4 ml). The mixture reacted at room temperature for 12 h; it was then poured into water and the yellow solid product was collected by filtration. The condensation product was placed in a mixture of a 10% aqueous NaOH solution (10 ml) and ethanol (2 ml), stirred at room temperature for 24 h, then acidified with 10% hydrochloric acid to pH = 2, and extracted four times with 10 ml ethyl acetate. The organic phase was dried with anhydrous sodium sulfate

overnight and the solvent removed by distillation under reduced pressure. The pale-brown solid residue was dissolved in ethanol at room temperature. Colorless triclinic crystals suitable for X-ray analysis (m.p. 443 K, 84.6% yield) grew over a period of 10 d when the solution was exposed to air. ^{1}H NMR: δ 12.21 (*brs*, 1H), 8.11 (*t*, 1H), 7.06 (*d*, 1H), 6.80 (*d*, 1H), 3.79 (*s*, 3H), 3.34 (*m*, 2H), 2.45 (*t*, 2H); IR(KBr): 3403, 2954, 1710, 1607, 1552, 1513, 1414, 1203. Elemental analysis calculated for $C_9H_{11}BrN_2O_3$: C 39.29, H 4.03, N 10.18%; found: C 39.43, H 3.95, N 10.11%.

Crystal data

$C_9H_{11}BrN_2O_3$	$Z = 2$
$M_r = 275.11$	$D_x = 1.717$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.824$ (4) Å	Cell parameters from 783 reflections
$b = 8.153$ (4) Å	$\theta = 2.3$–$27.0°$
$c = 9.227$ (4) Å	$\mu = 3.85$ mm^{-1}
$\alpha = 102.202$ (8)°	$T = 293$ (2) K
$\beta = 97.123$ (8)°	Block, colorless
$\gamma = 108.950$ (8)°	0.50 × 0.46 × 0.27 mm
$V = 532.2$ (4) Å^3	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	1763 independent reflections
	1644 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.018$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -8 \rightarrow 9$
$T_{min} = 0.208$, $T_{max} = 0.354$	$k = -9 \rightarrow 9$
2812 measured reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 0.2132P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.036$	
$wR(F^2) = 0.100$	
$S = 1.10$	$(\Delta/\sigma)_{max} = 0.001$
1763 reflections	$\Delta\rho_{max} = 0.84$ e Å^{-3}
141 parameters	$\Delta\rho_{min} = -0.62$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.011 (4)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1···O1^{i}	0.77 (5)	1.88 (5)	2.632 (3)	163 (5)
N2—H2*B*···O2^{ii}	0.86	2.28	3.005 (3)	142

Symmetry codes: (i) $1 + x, y, z$; (ii) $2 - x, 2 - y, 2 - z$.

The H atom attached to O3 was located in a difference Fourier map and refined as riding, with the isotropic displacement parameter allowed to vary freely. All other H atoms were positioned geometrically (C—H = 0.96 Å for CH_3, C—H = 0.97 Å for CH_2, 0.93 Å for CH and N—H = 0.86 Å) and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C,N)$, or $1.5U_{eq}$ for the methyl group.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

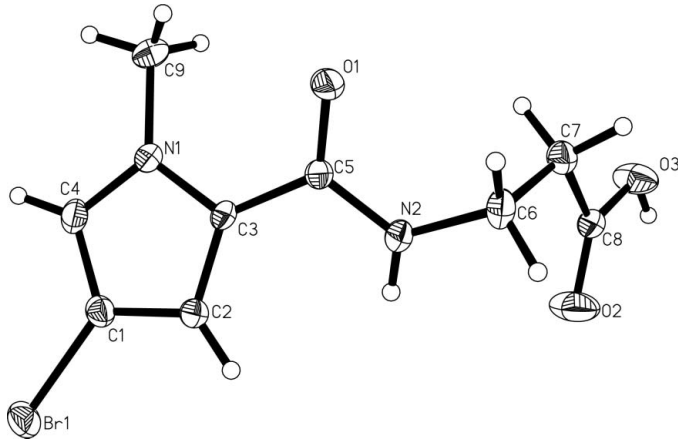


Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

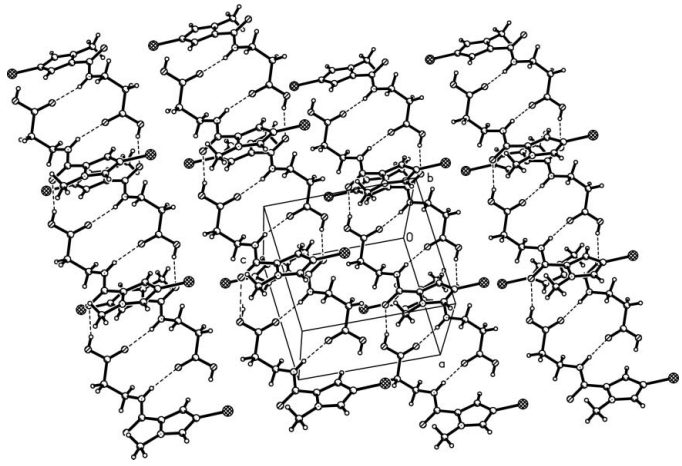


Figure 2
The packing of the title compound, showing the ribbons formed by hydrogen bonds (dashed lines).

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