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3-[(4,5-Dibromo-1-methyl-1*H*-pyrrole-2-carbonyl)-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 = 0.045
wR factor = 0.126
Data-to-parameter ratio = 14.8

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

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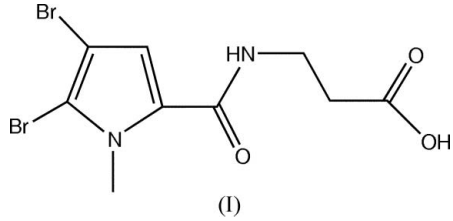
The title compound, $C_9H_{10}Br_2N_2O_3$, was synthesized by condensation of β-alanine methyl ester with 4,5-dibromo-1-methyl-2-trichloroacetylpyrrole at room temperature, followed by saponification and acidification. In the crystal structure, intermolecular N—H···O and O—H···O hydrogen-bond interactions 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 of them are bioactive substances (Tasdemir *et al.*, 2002). In our search for bioactive compounds, a series of brominated (pyrrole-2-carbonyl)amino acids and their methyl esters, including the title compound, (I), have been synthesized by reaction of β-alanine methyl ester with brominated 2-trichloroacetylpyrrole, or brominated 1-methyl-2-trichloroacetylpyrrole, followed by saponification and acidification. Pharmacological studies have shown that (I) inhibits *Streptococcus faecalis* and *Micrococcus luteus* moderately. We report here its crystal structure.



(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, molecules are linked through intermolecular N—H···O hydrogen bonds to give dimeric centrosymmetric $R_2^2(12)$ rings (Table 1), not $R_2^2(8)$ as in alanine (Liao *et al.*, 2001). 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 4,5-dibromo-1-methyl-2-(trichloroacetyl)pyrrole (1.92 g, 5 mmol) were added to acetonitrile (12 ml), followed by the addition of triethylamine (1.4 ml) dropwise. The mixture reacted at room temperature for 12 h, was then poured into water and the yellow solid product was collected after filtration. The condensation product was added to a mixture of 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. 458 K, in 69.0% yield) grew over a period of 7 d when the solution was exposed to air. ^{1}H NMR (acetone-d_6, 300 Hz): 6.09 (*s*, 1H), 3.93 (*s*, 3H), 3.53 (*t*, 2H), 2.59 (*t*, 2H); IR (KBr): 3396, 2961, 1716, 1607, 1550, 1506, 1419, 1196; elemental analysis calculated for $C_9H_{10}Br_2N_2O_3$: C 30.54, H 2.85, N 7.91%; found: C 30.43, H 2.95, N 7.72%.

Crystal data

$C_9H_{10}Br_2N_2O_3$
M_r = 354.01
Triclinic, $P\overline{1}$
a = 7.752 (2) Å
b = 9.238 (3) Å
c = 9.304 (3) Å
α = 104.062 (5)°
β = 107.942 (5)°
γ = 95.651 (5)°
V = 604.0 (3) Å^3
Z = 2
D_x = 1.947 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 816 reflections
θ = 3.3–27.0°
μ = 6.70 mm^{-1}
T = 273 (2) K
Block, colourless
0.50 × 0.38 × 0.20 mm
0.58 × 0.38 × 0.30 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi–scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.059, T_{max} = 0.262
5185 measured reflections
2602 independent reflections
2039 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.1°
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 11$
$l = -7 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.085
S = 1.02
2602 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 0.6113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.93 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1^{i}	0.82	1.86	2.663 (4)	167
N2—H2···O2^{ii}	0.86	2.32	3.128 (4)	157

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

The 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, N—H = 0.86 Å and O—H = 0.82 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,*N*), or 1.5U_{eq}(parent) for the methyl and hydroxy groups.

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

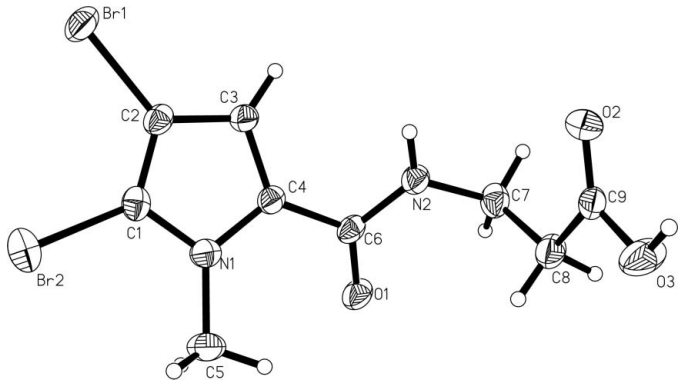


Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

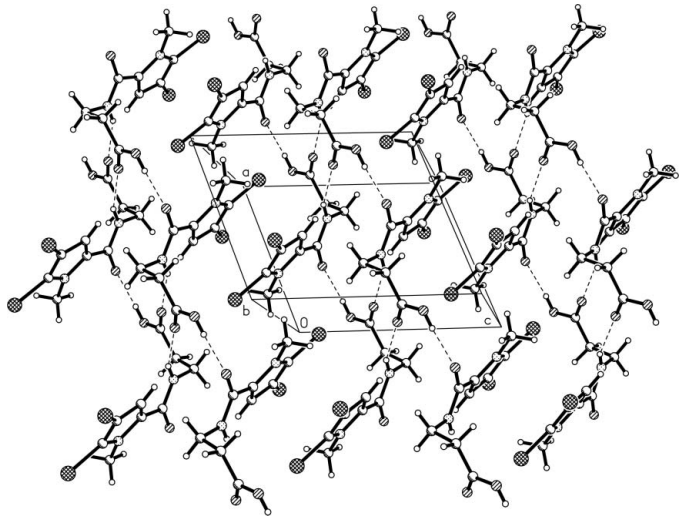


Figure 2
The crystal packing of the title compound, showing the ribbons formed by hydrogen bonds (dashed lines).

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