

Xiang-Chao Zeng,\* Shi-Hai Xu,  
Po-Run Liu and Jian GuDepartment of Chemistry, Jinan University,  
Guangzhou, Guangdong 510632, People's  
Republic of China

Correspondence e-mail: xczeng@sohu.com

## Key indicators

Single-crystal X-ray study  
 $T = 298\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$   
 $R$  factor = 0.038  
 $wR$  factor = 0.104  
Data-to-parameter ratio = 17.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.3-(4-Bromo-1*H*-pyrrole-2-carboxamido)-  
propanoic acid

The title compound,  $\text{C}_8\text{H}_9\text{BrN}_2\text{O}_3$ , was synthesized by condensation of  $\beta$ -alanine methyl ester with 4-bromo-2-(trichloroacetyl)pyrrole, followed by saponification and acidification. In the molecule, all bond lengths and angles are normal. The crystal packing is stabilized by intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds.

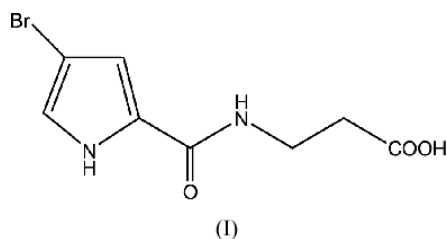
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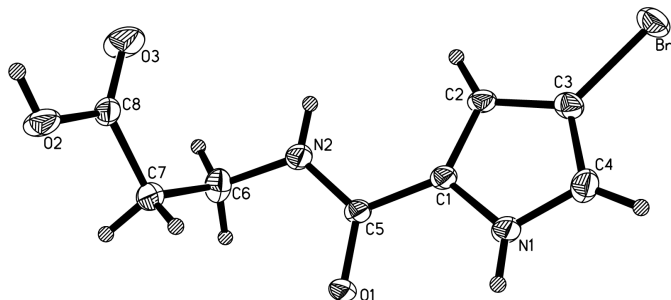
Online 25 March 2005

## Comment

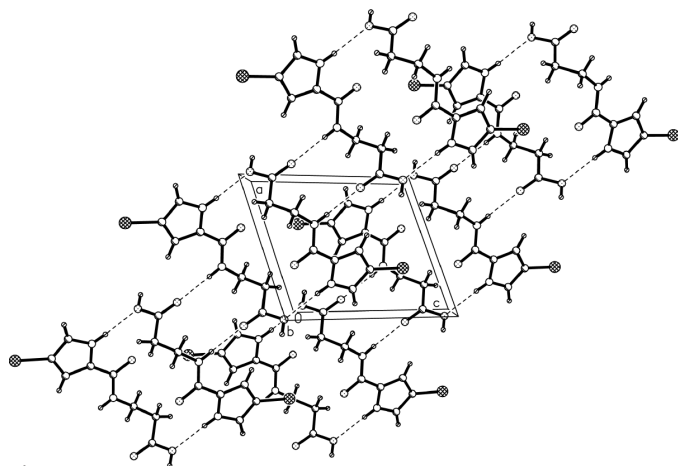
Pyrrole derivatives are well known in many marine organisms (Faulkner, 2001) and some are bioactive substances (Tasdemir *et al.*, 2002). In our search for bioactive compounds, a series of brominated (pyrrole-2-carbonyl)amino acids and their methyl esters had been synthesized by reaction of  $\beta$ -alanine methyl ester with 4-bromo-2-(trichloroacetyl)pyrrole or 4,5-dibromo-2-(trichloroacetyl)pyrrole followed by saponification and acidification. Among these compounds is the title compound, (I). Pharmacological studies have shown that (I) moderately inhibits *Streptococcus faecal* and *Micrococcus luteus*. We report here the crystal structure of (I).



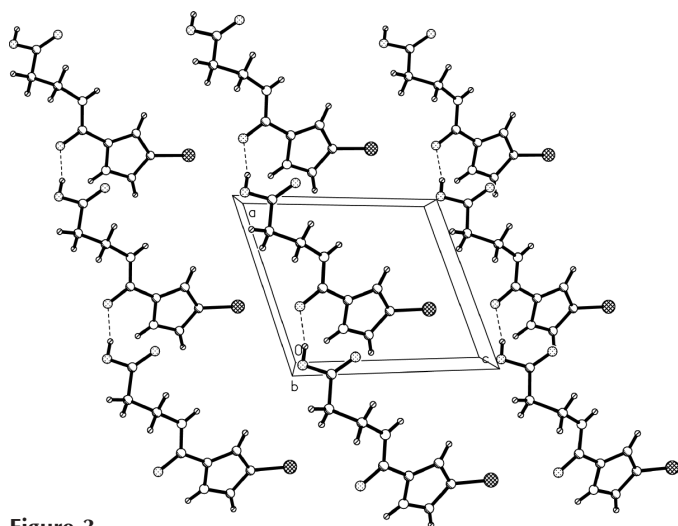
The bond lengths and angles in (I) are in good agreement with those in methyl (4,5-dibromo-1-methyl-1*H*-pyrrole-2-carboxylamino)acetate (Zeng *et al.*, 2004). In the crystal structure, there are three kinds of intermolecular hydrogen bonds (Table 1). The  $\text{N}2-\text{HN}2\cdots\text{O}3^{\text{ii}}$  hydrogen bond (symmetry codes as in Table 1) forms centrosymmetric dimers, which can be described as  $R_2^2(12)$ , compared with  $R_2^2(8)$



**Figure 1**  
View of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.



**Figure 2**  
N—H...O hydrogen-bonded (dashed lines) ribbons viewed along the *b* axis.

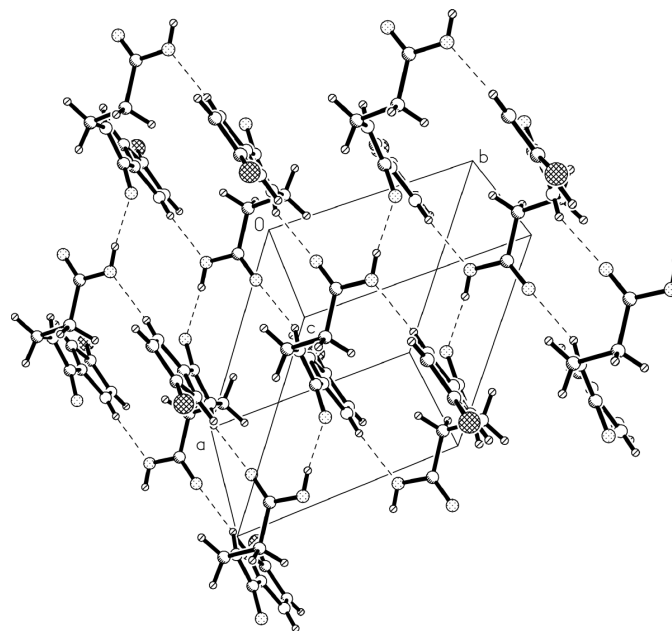


**Figure 3**  
O—H...O hydrogen-bonded (dashed lines) linear chains.

observed in bis( $\beta$ -alaninium) biphenyl-4,4'-disulfonatealanine (Liao *et al.*, 2001). An N1—HN1...O2<sup>1</sup> hydrogen bond forms another kind of dimer,  $R_2^2(18)$ . These intermolecular N—H...O hydrogen bonds generate a hydrogen-bonded ribbon (Fig. 2). At the same time, O—H...O (Table 1) hydrogen bonds link the molecules into linear chains along the *a* axis (Fig. 3), leading to the formation of extended two-dimensional layers (Fig. 4).

## Experimental

The hydrochloric acid salt of  $\beta$ -alanine methyl ester (0.70 g, 5 mmol) and 4-bromo-2-(trichloroacetyl)pyrrole (1.46 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 11 h, was then poured into water; the yellow solid product was collected after filtration. The condensation product was added to a mixture of a 10% NaOH water solution (10 ml) and ethanol (2 ml), stirred at room temperature for 24 h, and then acidified with 10% hydrochloric acid to a pH of 2 and extracted four times with 10 ml ethyl acetate. The organic phase was dried with anhydrous sodium



**Figure 4**  
Two-dimensional hydrogen-bonded (dashed lines) layers in (I).

sulfate overnight. The solvent was removed by distillation under reduced pressure and the pale-brown solid residue was collected. The crude product was dissolved in ethanol at room temperature and normal pressure. Pale-yellow block-like crystals suitable for X-ray analysis (m.p. 430 K, 79.3% yield) grew over a period of one week when the solution was exposed to air. <sup>1</sup>H NMR:  $\delta$  12.25 (*brs*, 1H), 11.79 (*brs*, 1H), 8.16 (*brs*, 1H), 6.95 (*m*, 1H), 6.81 (*m*, 1H), 3.38 (*m*, 2H), 2.47 (*t*, 2H); IR (KBr):  $\nu$  3385, 3344, 2959, 1725, 1581, 1529, 1419, 1340, 1197; elemental analysis calculated for C<sub>8</sub>H<sub>9</sub>BrN<sub>2</sub>O<sub>3</sub>: C 36.80, H 3.47, N 10.73%; found: C 36.91, H 3.29, N 10.61%.

### Crystal data

C<sub>8</sub>H<sub>9</sub>BrN<sub>2</sub>O<sub>3</sub>  
 $M_r = 261.07$   
Triclinic,  $P\bar{1}$   
 $a = 7.768$  (3) Å  
 $b = 8.497$  (3) Å  
 $c = 9.342$  (4) Å  
 $\alpha = 115.572$  (6)°  
 $\beta = 99.093$  (6)°  
 $\gamma = 106.548$  (6)°  
 $V = 504.2$  (3) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.720$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
Cell parameters from 946 reflections  
 $\theta = 2.89$ – $26.95^\circ$   
 $\mu = 4.06$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
Block, pale yellow  
0.50 × 0.45 × 0.20 mm

### Data collection

Bruker SMART 1K CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.151$ ,  $T_{\max} = 0.444$   
4276 measured reflections

2168 independent reflections  
1951 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$   
 $\theta_{\text{max}} = 27.0^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -10 \rightarrow 10$   
 $l = -11 \rightarrow 11$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.104$   
 $S = 1.05$   
2168 reflections  
127 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.3553P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.70$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.86$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—HN1 $\cdots$ O2 <sup>i</sup>	0.86	2.18	3.035 (2)	171
N2—HN2 $\cdots$ O3 <sup>ii</sup>	0.86	2.16	2.949 (2)	153
O2—H2A $\cdots$ O1 <sup>iii</sup>	0.82	1.81	2.617 (2)	167

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

The H atoms were positioned geometrically (C—H = 0.97 Å for CH<sub>2</sub> and 0.93 Å for CH, N—H = 0.86 Å and O—H = 0.82 Å) and refined using a riding model, with  $U_{iso} = 1.2U_{eq}$  of the parent atom.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINTE-Plus* (Bruker, 1999); data reduction: *SAINTE-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*.

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