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Ethyl (4-bromo-1*H*-pyrrole-2-carboxamido)acetate

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Key indicators

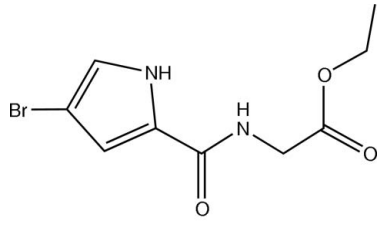
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.037
wR factor = 0.110
Data-to-parameter ratio = 18.5

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

The title compound, $C_9H_{11}BrN_2O_3$, was synthesized by condensation of glycine ethyl ester with 4-bromo-2-(trichloroacetyl)pyrrole at room temperature in 78.4% yield. In the crystal structure, intermolecular N—H···O hydrogen-bond interactions link the molecules into two-dimensional sheets.

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-carboxamido acid esters, including the title compound, (I), has been synthesized by reaction of amino acid esters with brominated 2-(trichloroacetyl)pyrrole, or brominated 1-methyl-2-(trichloroacetyl)-pyrrole.



(I)

We report here the structure of (I) which has been shown to inhibit *Streptococcus faecalis* and *Micrococcus luteus* moderately in pharmacological studies (Zeng *et al.*, 2004). Bond lengths and angles are unexceptional and are in good agreement with the corresponding values in [(4,5-dibromo-1-methyl-1*H*-pyrrole-2-carbonyl)amino]acetic acid methyl ester (Zeng *et al.*, 2004). In the crystal structure, there are two kinds of intermolecular hydrogen bonds. The N1—H···O1 hydrogen bonds (Table 1) form centrosymmetric dimers (Fig. 2), which graph-set analysis describes as an $R_2^2(10)$ motif (Bernstein *et al.*, 1995). At the same time, the dimeric units are further assembled into a herring-bone pattern in the overall

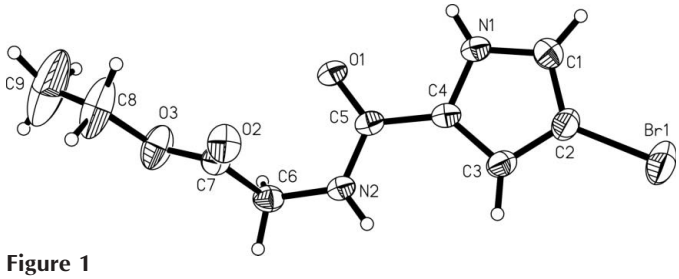


Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

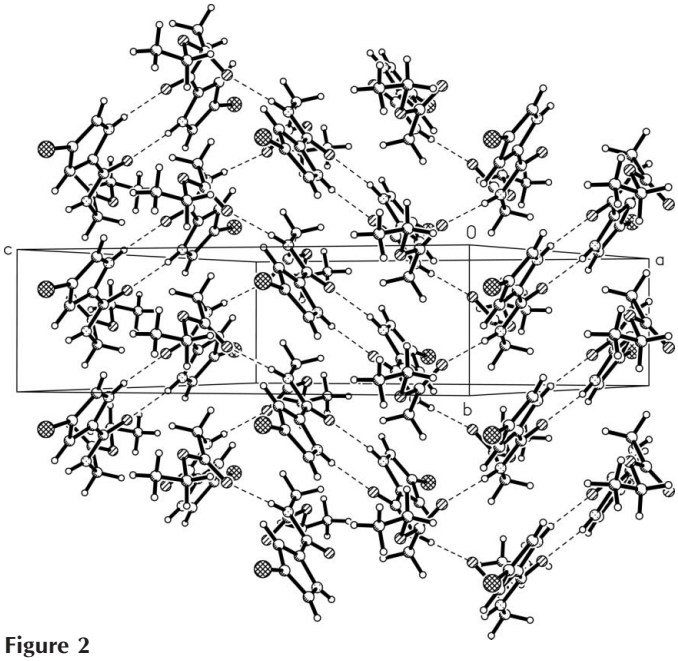


Figure 2
The crystal packing of (I), showing the two-dimensional sheet formed by hydrogen bonds (dashed lines).

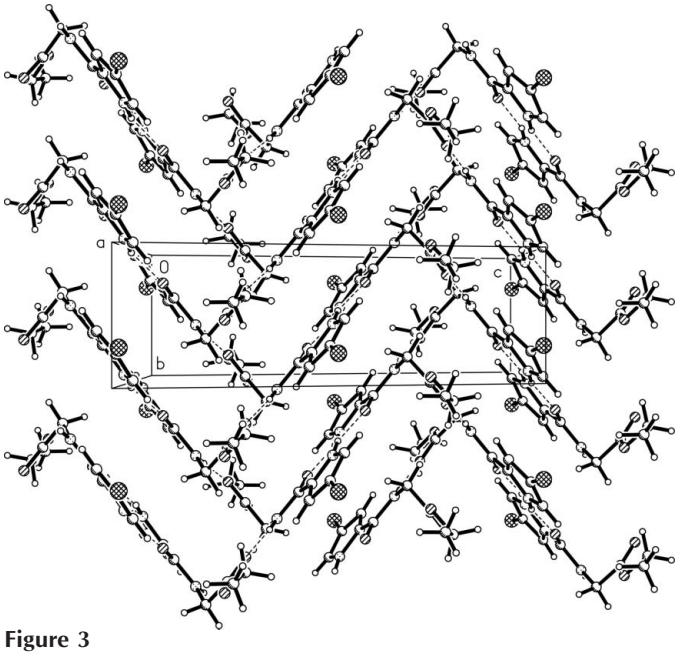


Figure 3
The crystal packing of (I). Dashed lines indicate hydrogen bonds.

crystal packing (Fig. 3). Individual dimeric units are linked by N2—H···O2 hydrogen bonds, generating two-dimensional sheets (Fig. 2).

Experimental

A hydrochloric acid salt of glycine ethyl 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 was stirred at room temperature for 10 h and then poured into water. After filtration, the precipitate was collected as a pale-yellow solid. The impure product was dissolved in ethanol at room temperature. Colorless monoclinic crystals suitable for X-ray analysis (m.p. 454 K, 78.4% yield) grew over a period of 7 d when the solution was exposed to air. ^{1}H NMR (DMSO-d_6, 300 Hz): 11.88 (*brs*, 1H), 8.57 (*t*, 1H), 7.01–6.99 (*m*, 1H), 6.87–6.86 (*m*, 1H), 3.96 (*d*, 2H), 3.53 (*q*, 2H), 1.21 (*t*, 3H); IR(KBr): 3375, 3217, 3140, 1732, 1644, 1566, 1524, 1335, 1224, 1130; analysis calculated for $C_9H_{11}BrN_2O_3$: C 39.29, H 4.03, N 10.18%; found: C 39.41, H 3.95, N 10.10%.

Crystal data

$C_9H_{11}BrN_2O_3$
M_r = 275.11
Monoclinic, $P2_1/c$
a = 14.944 (2) Å
b = 5.0501 (8) Å
c = 16.264 (3) Å
β = 107.843 (3)°
V = 1168.4 (3) Å^3
Z = 4
D_x = 1.564 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1926 reflections
θ = 2.6–26.3°
μ = 3.51 mm^{-1}
T = 298 (2) K
Block, colourless
0.50 × 0.38 × 0.34 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.202, T_{max} = 0.303
6732 measured reflections
2550 independent reflections
1562 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.0°
$h = -16 \rightarrow 19$
$k = -5 \rightarrow 6$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.110
S = 1.03
2550 reflections
138 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.5034P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0053 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	1.97	2.788 (3)	159
N2—H2···O2^{ii}	0.86	2.08	2.935 (3)	174

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

H atoms were positioned geometrically (C—H = 0.96 Å for CH_3, C—H = 0.97 Å for CH_2, C—H = 0.93 Å for CH and N—H = 0.86 Å) and refined using a riding model, with $U_{iso} = 1.2U_{eq}$ ($1.5U_{eq}$ for the methyl group) of the parent atom.

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*.

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References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Faulkner, D. J. (2001). *Nat. Prod. Rep.* **18**, 1–49.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tasdemir, D., Mallon, R., Greenstein. M., Feldberg, L. R., Kim, S. C., Collins, K., Wojciechowicz, D., Mangalindan, G. C., Concepcion, G. P., Harper, M. K. & Ireland, C. M. (2002). *J. Med. Chem.* **45**, 529–532.
Zeng, X. C., Xu, S. H., Deng, Q. Y., Cai, J. W., Guo, S. H., Gu, J. & He, D. H. (2004). *Acta Cryst.* E**60**, o1283–o1284.