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3-[(3,4,5-Tribromo-1*H*-pyrrol-2-ylcarbonyl)-amino]propanoic acid

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The title compound, $C_8H_7Br_3N_2O_3$, was synthesized by condensation of β-alanine methyl ester with 3,4,5-tribromo-2-trichloroacetylpyrrole at room temperature, followed by saponification and acidification. In the crystal structure, intermolecular N—H···O hydrogen bonds and weak O—H···Br hydrogen-bond interactions link the molecules into extended ribbons parallel to the *c* axis.

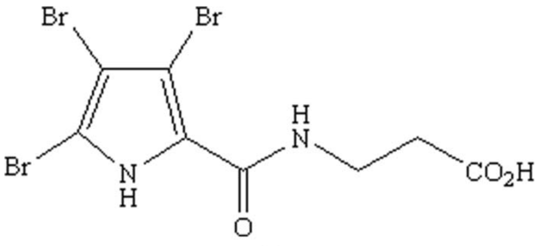
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.007 Å
R factor = 0.037
wR factor = 0.111
Data-to-parameter ratio = 15.9

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

Comment

Pyrrole derivatives are well known in many marine organisms (Faulkner, 2001); some are bioactive substances (Tasdemir *et al.*, 2002; Liu *et al.*, 2005). In our search for bioactive compounds, a series of (pyrrol-2-ylcarbonyl)amino acids and their esters, including the title compound, (I), have been synthesized by reaction of amino acid esters with 2-trichloroacetylpyrrole, or brominated 2-trichloroacetylpyrroles, followed by saponification and acidification. We report here the crystal structure of the title compound.



(I)

Bond lengths and angles are unexceptional and are in good agreement with the corresponding values in ethyl (3,4,5-tribromo-1*H*-pyrrole-2-carboxamido)acetate (Zeng *et al.*, 2006) and 3-(4-bromo-1*H*-pyrrole-2-carboxamido)propanoic acid (Zeng *et al.*, 2005).

In the crystal structure, there are two types of intermolecular hydrogen bonds (Table 1). Molecules are linked through N—H···O hydrogen bonds, forming centrosymmetric dimers (Fig. 2) of graph-set motif $R_2^2(10)$ (Bernstein *et al.*, 1995). The dimers are connected by weak O—H···Br hydrogen-bond interactions, generating ribbons running parallel to the *c* axis (also shown in Fig. 2).

Experimental

The hydrochloric acid salt of β-alanine methyl ester (0.70 g, 5 mmol) and 3,4,5-tribromo-2-trichloroacetylpyrrole (2.25 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

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12 h, was then poured into water and the yellow product was collected after separating the water. The condensation product was put into a mixture of 10% NaOH aqueous solution (10 ml) and ethanol (2 ml), stirred at room temperature for 24 h, then acidified with 10% hydrochloric acid to pH = 2, and extrtacted 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-yellow solid residue was dissolved in ethanol at room temperature. Colorless crystals suitable for X-ray analysis (m.p. 481 K, in 81.2% yield) grew over a period of 10 d when the solution was exposed to air.

Crystal data

$C_8H_7Br_3N_2O_3$
$M_r = 418.89$
Triclinic, $P\bar{1}$
$a = 7.0199$ (10) Å
$b = 8.0368$ (12) Å
$c = 11.5897$ (16) Å
$\alpha = 97.019$ (2)°
$\beta = 103.741$ (2)°
$\gamma = 102.156$ (2)°
$V = 610.61$ (15) Å^3
$Z = 2$
$D_x = 2.278$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 9.90$ mm^{-1}
$T = 273$ (2) K
Block, colorless
0.34 × 0.29 × 0.26 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick,1996)
$T_{\min} = 0.050$, $T_{\max} = 0.072$ (expected range = 0.053–0.076)
4506 measured reflections
2328 independent reflections
1837 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.111$
$S = 1.05$
2328 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.8415P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.80$ e Å^{-3}
$\Delta\rho_{\min} = -0.81$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···Br2^{i}	0.82	2.75	3.217 (4)	118
N1—H1···O1^{ii}	0.86	1.93	2.777 (5)	168

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

The H atoms were positioned geometrically (C—H = 0.97 Å for CH_2, O—H = 0.82 Å for OH and N—H = 0.86 Å) and refined using a riding model, with $U_{\rm iso}(H) = 1.2U_{\rm eq}$ 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: *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*.

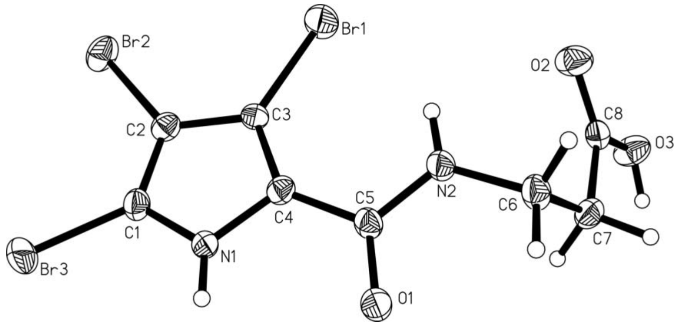

Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

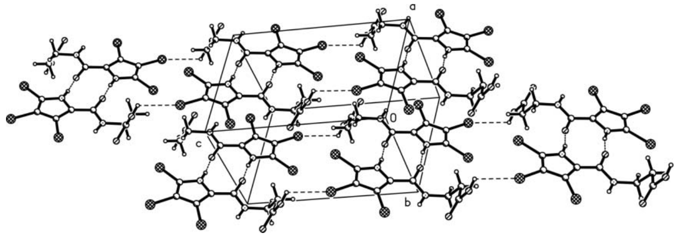

Figure 2
The packing of the title compound, showing the centrosymmetric dimers and the ribbons formed by hydrogen bonds (dashed lines).

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