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(*S*)-Methyl 2-(4,5-dibromo-1*H*-pyrrole-2-carboxamido)-3-methylbutanoate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.033
wR factor = 0.075
Data-to-parameter ratio = 19.1

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

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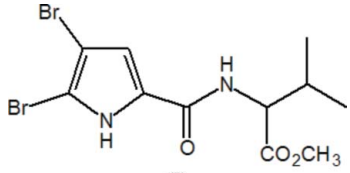
The title compound, $C_{11}H_{14}Br_2N_2O_3$, was synthesized by condensation of methyl l-2-amino-3-methylbutanoate with 4,5-dibromo-2-trichloroacetylpyrrole at room temperature. In the crystal structure, intermolecular N—H···O hydrogen-bonding interactions link the molecules, forming extended chains parallel to [010]. Bioactivity tests show that the title compound inhibits bacteria and also exhibits cytotoxicity.

Comment

Pyrrole derivatives are well known in many marine organisms (Faulkner, 2001), and some are known to be bioactive (Tasdemir *et al.*, 2002). In our search for bioactive compounds, a series of brominated pyrrole(2-carbonyl)amino acid esters, including the title compound, (I), has been synthesized by the reaction of amino acid esters with brominated 2-trichloroacetylpyrrole or with brominated 1-methyl-2-trichloroacetylpyrrole. Here, we report the crystal structure of (I) and its bioactivity in pharmacological studies.



(I)

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-carbonylaminoacetic acid methyl ester (Zeng *et al.*, 2004) and in (*S*)-methyl-4-methyl-2-(1*H*-pyrrole-2-carboxamido)pentanoate (Zeng *et al.*, 2005).

In the crystal structure of the title compound, there are two kinds of intermolecular hydrogen bonds (Table 1). Each molecule is connected to two other molecules by N—H···O hydrogen-bonding interactions, forming extended chains parallel to [010] (Fig. 2). This packing mode is different from that in the crystal structure of (*S*)-methyl-4-methyl-2-(1*H*-pyrrole-2-carboxamido)pentanoate (MMP), which contains four unique intermolecular hydrogen bonds.

Preliminary antibiotic tests performed *in vitro* and determined by the agar dilution method (Feng, 2000) indicate that the title compound inhibits five bacteria. Antibiotic activities against these bacteria [determined as minimum inhibitory concentration (mg ml^{-1}) values] are as follows: *Streptococcus faecalis*, 0.078; *Salmonella choleraesu*, 0.156; *Micrococcus luteus*, 0.156; *Staphylococcus aureu*s, 0.156; and *Escherichia coli*, 0.156. The bioactivities are promising and warrant further studies of this type of compound.

Experimental

The hydrochloride salt of L-valine methyl ester (0.84 g, 5 mmol) and 4,5-dibromo-2-trichloroacetylpyrrole (1.85 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 8 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. Colourless plates suitable for X-ray analysis (m.p. 452 K; yield 80.9%) crystallized over a period of 7 d. ^{1}H NMR ($CDCl_3$, 300 Hz, δ, p.p.m.): 11.13 (*brs*, 1H), 6.65 (*d*, 1H), 6.44 (*d*, 1H), 4.74–4.70 (*m*, 1H), 3.70 (*s*, 3H), 2.24–2.13 (*m*, 1H), 0.94–0.88 (*m*, 6H); IR (KBr, ν, cm^{-1}): 3378, 3277, 3116, 1724, 1635, 1559, 1518, 1319, 1216, 1150; Elemental analysis, calculated for $C_{11}H_{14}Br_2N_2O_3$: C 34.58, H 3.69, N 7.33%; found: C 34.35, H 3.77, N 7.52%.

Crystal data

$C_{11}H_{14}Br_2N_2O_3$
M_r = 382.04
Orthorhombic, $P2_12_12_1$
a = 9.196 (3) Å
b = 10.928 (3) Å
c = 14.244 (4) Å
V = 1431.5 (7) Å^3
Z = 4
D_x = 1.773 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 905 reflections
θ = 2.6–24.8°
μ = 5.66 mm^{-1}
T = 293 (2) K
Plate, colourless
0.47 × 0.41 × 0.18 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.087, T_{max} = 0.361
8925 measured reflections
3112 independent reflections
2519 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.0°
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 1.03
3112 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2 + 0.4813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter: 0.020 (12), with 1313 Friedel pairs

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.86	2.58	3.437 (4)	176
N1—H1A···O2^{ii}	0.86	2.08	2.929 (4)	168

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

All non-H atoms were refined with anisotropic displacement parameters. The H atoms were positioned geometrically, with C—H = 0.98 Å for CH, 0.96 Å for CH_3 and C—H = 0.93 Å for CH(aromatic), 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*.

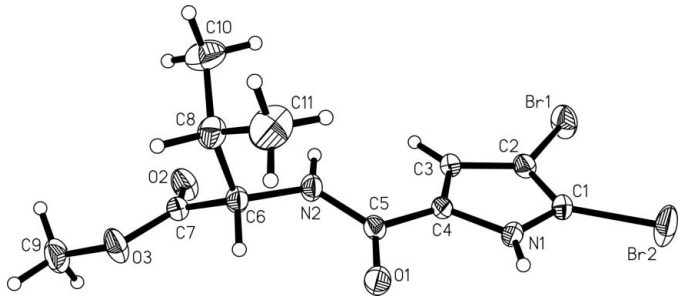


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

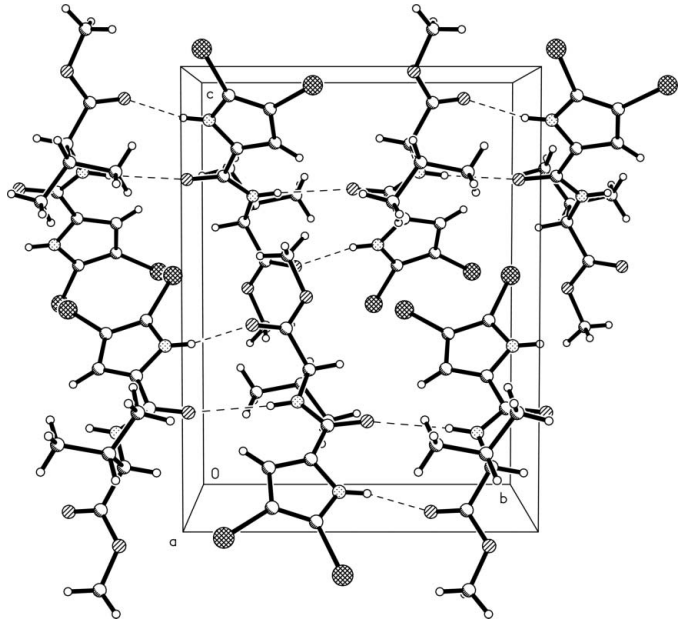


Figure 2
The packing of (I), showing the hydrogen-bonded chains (dashed lines).

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