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

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Key indicators

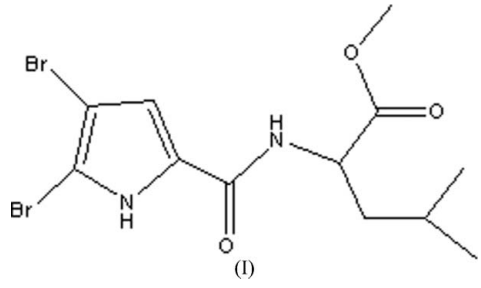
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.020 Å
R factor = 0.054
wR factor = 0.173
Data-to-parameter ratio = 16.3

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

The title compound, $C_{12}H_{16}Br_2N_2O_3$, synthesized by the condensation of L-leucine methyl ester with 4,5-dibromo-2-trichloroacetylpyrrole at room temperature, crystallizes with two independent molecules in the asymmetric unit. Intermolecular N—H···O hydrogen bonds link the molecules into ribbons extending along the *b* axis.

Comment

Pyrrole derivatives are well known as bioactive substances (Tasdemir *et al.*, 2002; Liu *et al.*, 2005). Examples are found in many marine organisms (Faulkner, 2001). In our search for bioactive compounds, a series of pyrrole(2-carbonyl)amino acid esters has been synthesized by the reaction of amino acid esters with 2-trichloroacetylpyrrole or brominated 2-trichloroacetylpyrroles. We report here the crystal structure of the title compound, (I).



(I)

Compound (I) crystallizes with two independent molecules in the asymmetric unit (Fig. 1). The bond lengths and angles in both molecules are unexceptional, being in good agreement with those observed in (*S*)-methyl 4-methyl-2-(1*H*-pyrrole-2-carboxamido)pentanoate (Zeng & Liu, 2005) and (*S*)-methyl 2-(4,5-dibromo-1*H*-pyrrole-2-carboxamido)-3-methylbutanoate (Zeng, 2006).

The crystal packing of (I) (Fig. 2) is stabilized by intermolecular N—H···O hydrogen bonds (Table 1), which link the molecules into ribbons extending along the *b* axis.

Experimental

The hydrochloric acid salt of L-leucine methyl ester (0.91 g, 5 mmol) and 4,5-dibromo-2-trichloroacetylpyrrole (1.85 g, 5 mmol) were added to acetonitrile (12 ml), and then triethylamine (1.4 ml) was added dropwise. The mixture was stirred at room temperature for 9 h and then poured into water. After filtration, the precipitate was collected as a light-yellow solid. The impure product was dissolved in 95% ethanol at room temperature. Colourless orthorhombic crystals of (I) suitable for X-ray analysis (m.p. 437 K; 86.9% yield) grew over a period of one week when the solution was exposed to air.

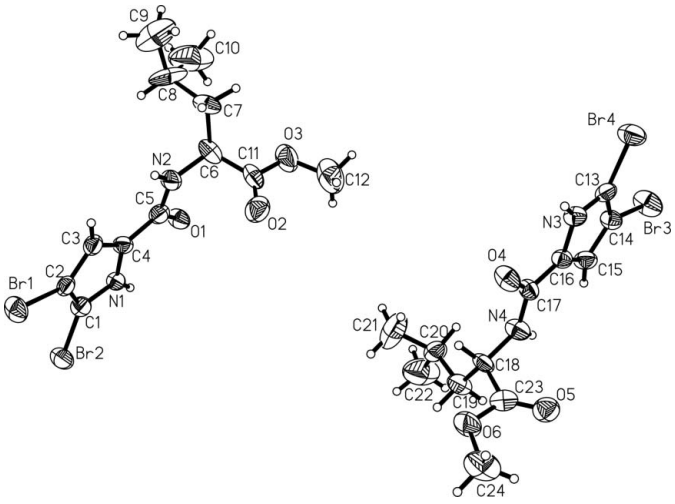


Figure 1
The two independent molecules of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

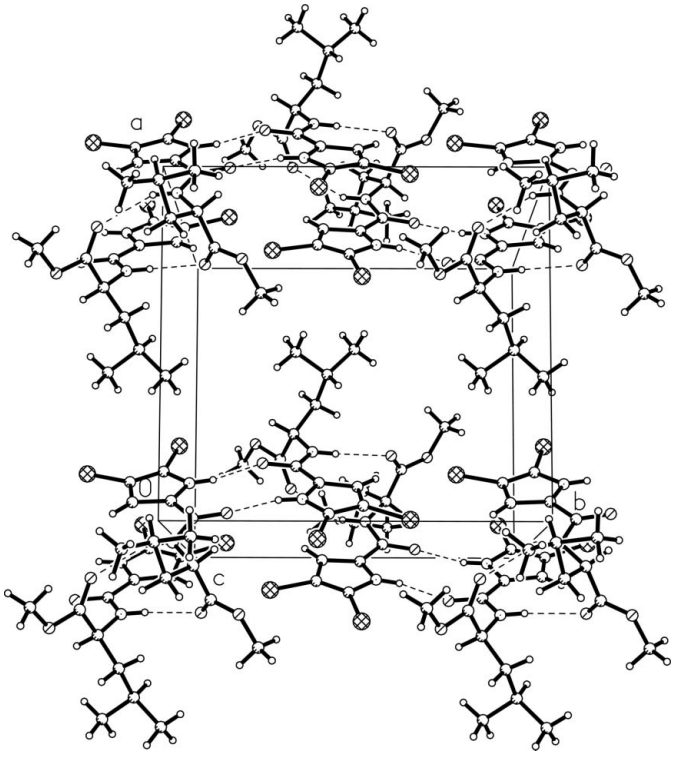


Figure 2
The crystal packing of (I), showing the intermolecular hydrogen bonds as dashed lines.

Crystal data

$C_{12}H_{16}Br_2N_2O_3$
M_r = 396.09
Orthorhombic, $P2_12_12_1$
a = 12.402 (2) Å
b = 13.479 (3) Å
c = 19.440 (4) Å
V = 3249.8 (11) Å^3
Z = 8
D_x = 1.619 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 4.99 mm^{-1}
T = 293 (2) K
Block, colourless
0.44 × 0.27 × 0.19 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.217, T_{max} = 0.451
(expected range = 0.187–0.387)
15481 measured reflections
5694 independent reflections
2490 reflections with $I > 2\sigma(I)$
R_{int} = 0.086
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.173
S = 1.02
5694 reflections
349 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 3.8188P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Absolute structure: Flack (1983), 2473 Friedel pairs
Flack parameter: 0.04 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O2^{i}	0.86	2.40	3.201 (16)	155
N3—H3A···O1^{ii}	0.86	1.98	2.819 (14)	164
N2—H2···O5^{iii}	0.86	2.28	3.068 (17)	153
N1—H1···O4^{iv}	0.86	1.97	2.804 (13)	163

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

H atoms were positioned geometrically, with C—H = 0.93–0.98 Å and N—H = 0.86 Å, and refined using a riding model, with U_{iso}(H) = 1.2–1.5 times U_{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*.

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