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(*S*)-Methyl 4-methyl-2-(1*H*-pyrrole-2-carboxamido)pentanoate

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Key indicators

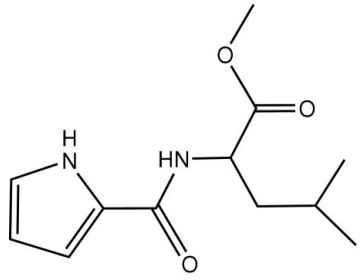
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.120
Data-to-parameter ratio = 10.5

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

The title compound, $C_{12}H_{18}N_2O_3$, was synthesized in 87.2% yield by condensation of L-leucine methyl ester with 2-trichloroacetylpyrrole at room temperature. There are two chemically equivalent and crystallographically independent molecules in the asymmetric unit. In the crystal structure, intermolecular N—H···O hydrogen-bonding interactions link the molecules into extended chains parallel to the *c* axis.

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 pyrrole(2-carbonyl)amino acid esters, including the title compound, (I), have been synthesized by the reaction of amino acid esters with 2-trichloroacetylpyrrole or brominated 2-trichloroacetylpyrroles. Pharmacological studies have shown that the title compound, (I), moderately inhibits *Streptococcus faecalis* and *Micrococcus luteus*. We report here its crystal structure.



(I)

Bond lengths and angles are unexceptional and are in good agreement with the corresponding values in 3-(pyrrole-2-carboxamido)propanoic acid(Zeng *et al.*, 2005) and methyl (4,5-dibromo-1-methyl-1*H*-pyrrole-2-carbonylamino)acetate (Zeng *et al.*, 2004).

There are two molecules in the asymmetric unit (Fig. 1) and four kinds of intermolecular hydrogen bonds (Table 1) in the crystal structure. Every molecule is connected with two other molecules by four N—H···O hydrogen-bond interactions, generating extended chains along the *c* axis (Fig. 2).

Experimental

The hydrochloric acid salt of L-leucine methyl ester (0.91 g, 5 mmol) and 2-trichloroacetylpyrrole (1.06 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 12 h and then poured into water. After filtration, the precipitate was collected as a

pale-brown solid. The impure product was dissolved in ethanol at room temperature. Colorless orthorhombic crystals suitable for X-ray analysis (m.p. 408 K, 87.2% yield) grew over a period of one week when the solution was exposed to air. ^{1}H NMR ($CDCl_3$, 500 Hz): 10.02 (*brs*, 1H), 6.92–6.90 (*m*, 1H), 6.66–6.65 (*m*, 1H), 6.47 (*brs*, 1H), 6.22–6.20 (*m*, 1H), 4.85–4.81 (*m*, 1H), 3.74 (*s*, 3H), 1.77–1.63 (*m*, 3H), 0.99–0.95 (*m*, 6H); IR (KBr): 3373, 3277, 3066, 1732, 1627, 1558, 1521, 1356, 1201, 1120. Analysis calculated for $C_{12}H_{18}N_2O_3$: C 60.49, H 7.61, N 11.76%; found: C 60.35, H 7.51, N 11.74%.

Crystal data

$C_{12}H_{18}N_2O_3$
M_r = 238.28
Orthorhombic, $P2_12_12_1$
a = 8.920 (4) Å
b = 16.282 (8) Å
c = 18.504 (9) Å
V = 2688 (2) Å^3
Z = 8
D_x = 1.178 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 941 reflections
θ = 2.5–21.9°
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, colorless
0.50 × 0.26 × 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} = 0.974, T_{max} = 0.985
14949 measured reflections
3301 independent reflections
2196 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.1°
$h = -11 \rightarrow 10$
$k = -18 \rightarrow 20$
$l = -21 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.120
S = 1.01
3301 reflections
314 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2 + 0.3014P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O5	0.86	2.23 (1)	3.078 (3)	170 (1)
N2—H2···O4^{i}	0.86	2.14 (1)	2.935 (3)	154 (1)
N3—H3···O2^{i}	0.86	2.35 (1)	3.054 (3)	140 (1)
N4—H4···O1	0.86	2.05 (1)	2.861 (3)	158 (1)

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

The H atoms were positioned geometrically [C—H = 0.98 Å for CH, 0.97 Å for CH_2, 0.96 Å for CH_3 and 0.93 Å for CH(aromatic), and N—H = 0.86 Å] and refined using a riding model, with U_{iso}(H) values of 1.2U_{eq} (1.5U_{eq} for the methyl group) of the parent atom. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before refinement of the structure.

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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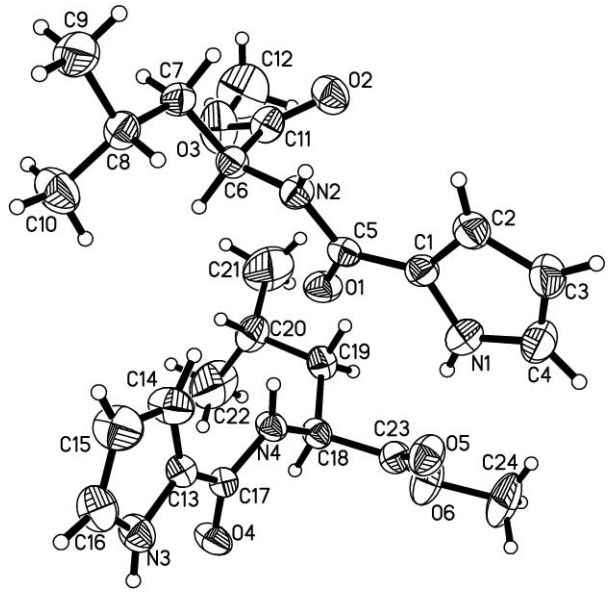

Figure 1
The asymmetric unit of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

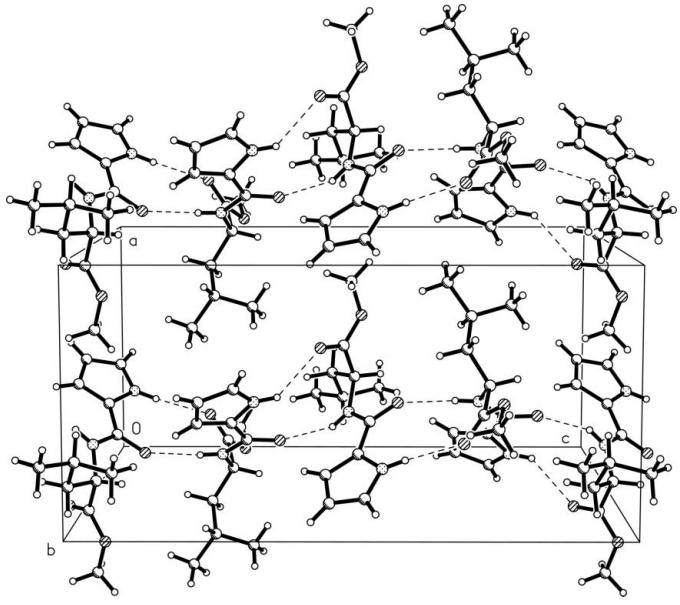

Figure 2
Crystal packing of the title compound showing the chains formed by hydrogen bonds (dashed lines).

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