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Methyl 3-(3,4,5-tribromo-1*H*-pyrrol-2-ylcarboxamido)propionate

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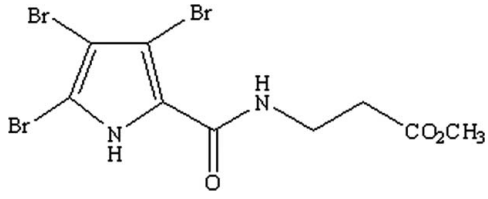
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.007 Å; R factor = 0.037; wR factor = 0.107; data-to-parameter ratio = 16.0.

In the crystal structure of the title compound, $C_9H_9Br_3N_2O_3$, molecules are linked by N—H···O hydrogen bonds to form centrosymmetric dimers of graph-set motif $R_2^2(10)$.

Related literature

For related literature, see: Banwell *et al.* (2006); Bernstein *et al.* (1995); Faulkner (2002); Sosa *et al.* (2002); Zeng (2006); Zeng *et al.* (2006).



Experimental

Crystal data

$C_9H_9Br_3N_2O_3$
M_r = 432.91
Monoclinic, $P2_1/c$
a = 8.7032 (12) Å
b = 8.6376 (12) Å
c = 16.962 (2) Å
β = 95.341 (2)°
V = 1269.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 9.53 mm^{-1}
T = 173 (2) K
0.44 × 0.31 × 0.25 mm

Data collection

Bruker SMART 1 K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.040, T_{max} = 0.092
7442 measured reflections
2484 independent reflections
2097 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.107
S = 1.14
2484 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −1.86 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88	1.89	2.751 (6)	166

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

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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2289).

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supplementary materials

Acta Cryst. (2007). E**63**, o2918 [**doi:10.1107/S1600536807022404**]

Methyl 3-(3,4,5-tribromo-1*H*-pyrrol-2-ylcarboxamido)propionate

X.-C. Zeng, X. Ling, J. Zeng and X. Li

Comment

Pyrrole derivatives are well known in many marine organisms (Faulkner, 2002), some show important bioactivities, such as antitumor activity (Banwell *et al.*, 2006) and protein kinase inhibiting activity (Sosa *et al.*, 2002). This is the reason why they have attracted our interest. This study follows our previous studies on 3-[(3,4,5-tribromo-1*H*-pyrrol-2-ylcarbonyl)amino]propanoic acid (Zeng *et al.*, 2006) and 3-bromo-1-methyl-6,7-dihydropyrrolo[2,3-*c*]azepine-4,8(1*H*,5H)-dione (Zeng, 2006).

In the crystal structure, molecules of the title compound are linked through N—H···O hydrogen bonds (Table 1) to form centrosymmetric dimers (Fig. 2) of graph-set motif $R_2^2(10)$ (Bernstein *et al.*, 1995). Bond lengths and angles are unexceptional.

Experimental

The hydrochloric acid salt of beta-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 was stirred at room temperature for 12 h and then poured into water. After filtration, the precipitate was collected as a yellow solid. The impure product was dissolved in EtOH at room temperature. Colourless monoclinic crystals suitable for X-ray analysis (m.p. 456 K, 90.3% yield) grew over a period of one week when the solution was exposed to the air. Analysis calculated for $C_9H_9Br_3N_2O_3$: C 24.97, H 2.09, N 6.47%. Found: C 24.82, H 2.15, N 6.54%.

Refinement

The H atoms were positioned geometrically [C—H = 0.99 Å for CH_2, 0.98 Å for CH_3, and N—H = 0.88 Å] and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}$ ($1.5U_{eq}$ for the methyl group) of the parent atom.

Figures

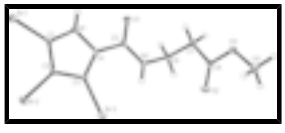

Fig. 1. The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. Dimers formed by hydrogen bonds (dashed lines).

Methyl 3-(3,4,5-tribromo-1*H*-pyrrol-2-ylcarboxamido)propionate

Crystal data

$C_9H_9Br_3N_2O_3$	F_{000} = 824
M_r = 432.91	D_x = 2.265 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 456 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.7032 (12) Å	Cell parameters from 2696 reflections
b = 8.6376 (12) Å	θ = 2.6–22.7º
c = 16.962 (2) Å	μ = 9.53 mm^{-1}
β = 95.341 (2)º	T = 173 (2) K
V = 1269.6 (3) Å^3	Prism, colourless
Z = 4	0.44 × 0.31 × 0.25 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer	2484 independent reflections
Radiation source: fine-focus sealed tube	2097 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 173(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −10→10
T_{min} = 0.040, T_{max} = 0.092	k = −9→10
7442 measured reflections	l = −18→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H-atom parameters constrained
$wR(F^2)$ = 0.107	$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 6.1964P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.14	$(\Delta/\sigma)_{max}$ = 0.001
2484 reflections	$\Delta\rho_{max}$ = 0.77 e Å^{-3}
155 parameters	$\Delta\rho_{min}$ = −1.86 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br2	0.33602 (6)	0.27122 (7)	0.89835 (3)	0.02380 (18)
Br1	0.61311 (6)	0.24109 (7)	1.07246 (3)	0.02411 (18)
C9	1.1909 (7)	0.4604 (9)	0.5581 (4)	0.0341 (15)
H9A	1.1514	0.5392	0.5199	0.051*
H9B	1.1172	0.3746	0.5579	0.051*
H9C	1.2902	0.4215	0.5434	0.051*
C4	0.7555 (6)	0.4767 (6)	0.8898 (3)	0.0157 (10)
C3	0.6101 (6)	0.4372 (7)	0.8553 (3)	0.0164 (11)
O1	1.0017 (4)	0.5867 (5)	0.9124 (2)	0.0272 (10)
N2	0.8719 (5)	0.6408 (5)	0.7957 (3)	0.0176 (9)
H2	0.7916	0.6209	0.7618	0.021*
N1	0.7698 (5)	0.4137 (5)	0.9641 (3)	0.0168 (9)
H1	0.8523	0.4207	0.9981	0.020*
O3	1.2117 (4)	0.5281 (5)	0.6367 (2)	0.0260 (9)
C6	0.9914 (6)	0.7501 (6)	0.7736 (3)	0.0188 (11)
H6A	1.0287	0.8119	0.8207	0.023*
H6B	0.9446	0.8224	0.7329	0.023*
O2	0.9712 (4)	0.6185 (5)	0.6209 (2)	0.0293 (10)
C1	0.6386 (6)	0.3391 (6)	0.9774 (3)	0.0156 (11)
C7	1.1278 (6)	0.6691 (6)	0.7418 (3)	0.0170 (11)
H7A	1.2142	0.7435	0.7411	0.020*
H7B	1.1623	0.5836	0.7781	0.020*
C2	0.5355 (6)	0.3512 (6)	0.9103 (3)	0.0157 (11)
C8	1.0915 (6)	0.6051 (6)	0.6604 (3)	0.0174 (11)
C5	0.8841 (6)	0.5724 (6)	0.8659 (3)	0.0170 (11)
Br3	0.51990 (6)	0.48619 (7)	0.75407 (3)	0.02589 (18)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br2	0.0114 (3)	0.0376 (4)	0.0222 (3)	−0.0095 (2)	0.0003 (2)	−0.0027 (2)
Br1	0.0206 (3)	0.0334 (4)	0.0184 (3)	−0.0073 (2)	0.0021 (2)	0.0047 (2)

C9	0.028 (3)	0.050 (4)	0.025 (3)	0.007 (3)	0.004 (3)	−0.017 (3)
C4	0.011 (2)	0.022 (3)	0.014 (2)	0.001 (2)	0.0006 (18)	0.002 (2)
C3	0.010 (2)	0.027 (3)	0.011 (2)	0.003 (2)	−0.0012 (19)	−0.001 (2)
O1	0.0143 (19)	0.044 (3)	0.022 (2)	−0.0086 (18)	−0.0054 (16)	0.0083 (19)
N2	0.011 (2)	0.026 (3)	0.015 (2)	−0.0033 (18)	0.0004 (17)	0.0028 (19)
N1	0.009 (2)	0.021 (2)	0.019 (2)	−0.0011 (17)	−0.0033 (17)	−0.0017 (19)
O3	0.0174 (19)	0.041 (3)	0.019 (2)	0.0071 (18)	0.0013 (15)	−0.0054 (18)
C6	0.016 (3)	0.020 (3)	0.021 (3)	−0.004 (2)	0.003 (2)	0.003 (2)
O2	0.019 (2)	0.045 (3)	0.022 (2)	0.0105 (19)	−0.0062 (16)	−0.0085 (19)
C1	0.011 (2)	0.020 (3)	0.015 (3)	−0.002 (2)	0.0006 (19)	−0.001 (2)
C7	0.012 (2)	0.019 (3)	0.021 (3)	−0.001 (2)	0.003 (2)	0.001 (2)
C2	0.008 (2)	0.021 (3)	0.018 (3)	−0.002 (2)	0.0002 (19)	−0.003 (2)
C8	0.014 (3)	0.021 (3)	0.018 (3)	−0.001 (2)	0.004 (2)	0.005 (2)
C5	0.008 (2)	0.021 (3)	0.022 (3)	−0.002 (2)	0.002 (2)	−0.003 (2)
Br3	0.0185 (3)	0.0375 (4)	0.0206 (3)	0.0007 (2)	−0.0034 (2)	0.0017 (2)

Geometric parameters (Å, °)

Br2—C2	1.862 (5)	N2—C6	1.479 (7)
Br1—C1	1.852 (5)	N2—H2	0.8800
C9—O3	1.453 (7)	N1—C1	1.349 (7)
C9—H9A	0.9800	N1—H1	0.8800
C9—H9B	0.9800	O3—C8	1.332 (6)
C9—H9C	0.9800	C6—C7	1.520 (7)
C4—N1	1.368 (7)	C6—H6A	0.9900
C4—C3	1.386 (7)	C6—H6B	0.9900
C4—C5	1.478 (7)	O2—C8	1.196 (6)
C3—C2	1.398 (7)	C1—C2	1.387 (7)
C3—Br3	1.869 (5)	C7—C8	1.493 (7)
O1—C5	1.239 (6)	C7—H7A	0.9900
N2—C5	1.324 (7)	C7—H7B	0.9900
O3—C9—H9A	109.5	N2—C6—H6B	109.0
O3—C9—H9B	109.5	C7—C6—H6B	109.0
H9A—C9—H9B	109.5	H6A—C6—H6B	107.8
O3—C9—H9C	109.5	N1—C1—C2	108.6 (5)
H9A—C9—H9C	109.5	N1—C1—Br1	122.5 (4)
H9B—C9—H9C	109.5	C2—C1—Br1	128.9 (4)
N1—C4—C3	107.0 (4)	C8—C7—C6	113.2 (4)
N1—C4—C5	117.9 (4)	C8—C7—H7A	108.9
C3—C4—C5	135.0 (5)	C6—C7—H7A	108.9
C4—C3—C2	108.2 (4)	C8—C7—H7B	108.9
C4—C3—Br3	128.4 (4)	C6—C7—H7B	108.9
C2—C3—Br3	123.5 (4)	H7A—C7—H7B	107.7
C5—N2—C6	120.9 (4)	C1—C2—C3	106.4 (4)
C5—N2—H2	119.6	C1—C2—Br2	125.9 (4)
C6—N2—H2	119.6	C3—C2—Br2	127.7 (4)
C1—N1—C4	109.8 (4)	O2—C8—O3	123.4 (5)
C1—N1—H1	125.1	O2—C8—C7	125.5 (5)
C4—N1—H1	125.1	O3—C8—C7	111.1 (4)

C8—O3—C9	116.4 (4)	O1—C5—N2	121.3 (5)
N2—C6—C7	112.8 (4)	O1—C5—C4	118.9 (5)
N2—C6—H6A	109.0	N2—C5—C4	119.8 (4)
C7—C6—H6A	109.0		
N1—C4—C3—C2	0.9 (6)	C4—C3—C2—C1	−0.4 (6)
C5—C4—C3—C2	−175.4 (6)	Br3—C3—C2—C1	179.9 (4)
N1—C4—C3—Br3	−179.5 (4)	C4—C3—C2—Br2	178.3 (4)
C5—C4—C3—Br3	4.3 (10)	Br3—C3—C2—Br2	−1.4 (7)
C3—C4—N1—C1	−1.0 (6)	C9—O3—C8—O2	0.7 (8)
C5—C4—N1—C1	176.0 (5)	C9—O3—C8—C7	−179.9 (5)
C5—N2—C6—C7	83.9 (6)	C6—C7—C8—O2	2.8 (8)
C4—N1—C1—C2	0.7 (6)	C6—C7—C8—O3	−176.7 (4)
C4—N1—C1—Br1	−179.1 (4)	C6—N2—C5—O1	−6.5 (8)
N2—C6—C7—C8	73.0 (6)	C6—N2—C5—C4	173.5 (5)
N1—C1—C2—C3	−0.2 (6)	N1—C4—C5—O1	2.9 (8)
Br1—C1—C2—C3	179.7 (4)	C3—C4—C5—O1	178.8 (6)
N1—C1—C2—Br2	−178.9 (4)	N1—C4—C5—N2	−177.2 (5)
Br1—C1—C2—Br2	1.0 (8)	C3—C4—C5—N2	−1.2 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88	1.89	2.751 (6)	166

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+2.

Fig. 1

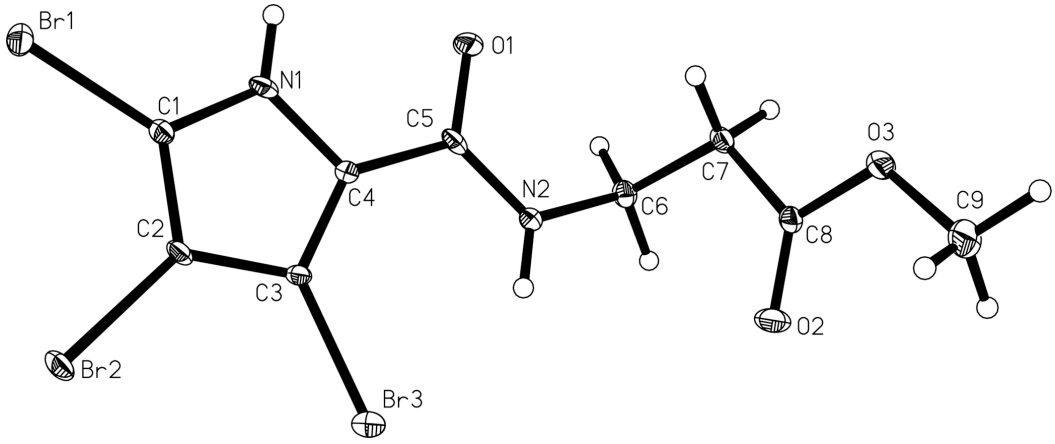

Fig. 2