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Ethyl (3,4,5-tribromo-1*H*-pyrrole-2-carboxamido)acetate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.036
wR factor = 0.099
Data-to-parameter ratio = 16.1

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

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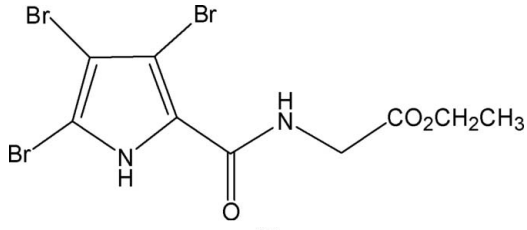
The title compound, $C_9H_9Br_3N_2O_3$, was synthesized by the condensation of ethyl aminoacetate with 3,4,5-tribromo-2-trichloroacetylpyrrole at room temperature. In the crystal structure, intermolecular N—H···O hydrogen-bond interactions form centrosymmetric dimers.

Comment

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



(I)

The bond lengths and angles are unexceptional and are in good agreement with the corresponding parameters in ethyl (4-bromo-1-*H*-pyrrole-2-carboxamido)acetate (Zeng, 2005).

In the crystal structure, there is one type of intermolecular hydrogen bond (Table 1). These N—H···O hydrogen bonds form centrosymmetric dimers (Fig. 2) of graph-set motif $R_2^2(10)$ (Bernstein *et al.*, 1995).

Experimental

A hydrochloric acid salt of glycine ethyl ester (0.70 g, 5 mmol) and 3,4,5-tribromo-2-trichloroacetylpyrrole (2.25 g, 5 mmol) was added to 10 ml of acetonitrile, 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 methanol at room temperature. Colourless crystals suitable for X-ray analysis (m. p. 495 K, yield 91.7%) grew over a period of ten days when the solution was exposed to air. ^{1}H NMR (DMSO-d_6, 300 Hz): 13.18 (*s*, 1H), 7.90 (*t*, *J* = 5.7, 1H), 4.11 (*q*, *J* = 7.2, 2H), 4.02 (*d*, *J* = 5.7, 2H), 1.20 (*t*, *J* = 7.2, 3H); IR(KBr): 3394, 3150, 1724, 1629, 1561, 1524, 1384, 1222; Analysis calculated for $C_9H_9Br_3N_2O_3$: C 24.97, H 2.09, N 6.47%; found: C 25.11, H 2.13, N 6.38%.

Crystal data

$C_9H_9Br_3N_2O_3$	$Z = 2$
$M_r = 432.91$	$D_x = 2.205$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.8767$ (13) Å	Cell parameters from 2525 reflections
$b = 9.2500$ (13) Å	$\theta = 2.3$–$27.0°$
$c = 9.4370$ (13) Å	$\mu = 9.28$ mm^{-1}
$\alpha = 71.890$ (2)°	$T = 293$ (2) K
$\beta = 63.062$ (2)°	Block, colourless
$\gamma = 76.166$ (2)°	0.40 × 0.31 × 0.29 mm
$V = 652.14$ (16) Å^3	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	2493 independent reflections
φ and ω scans	2009 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick,1996)	$R_{int} = 0.034$
$T_{min} = 0.044$, $T_{max} = 0.068$	$\theta_{max} = 26.0°$
4873 measured reflections	$h = -10 \rightarrow 10$
	$k = -11 \rightarrow 11$
	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.302P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.036$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.099$	$\Delta\rho_{max} = 0.68$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.72$ e Å^{-3}
2493 reflections	
155 parameters	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	1.94	2.773 (4)	161

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

H atoms were positioned geometrically [C—H = 0.97 Å for methylene H atoms, 0.96 Å for methyl H atoms, and N—H = 0.86 Å] and refined using a riding model, with $U_{iso} = 1.2U_{eq}$(C, N) or $1.5U_{eq}$(C) for methyl H atoms.

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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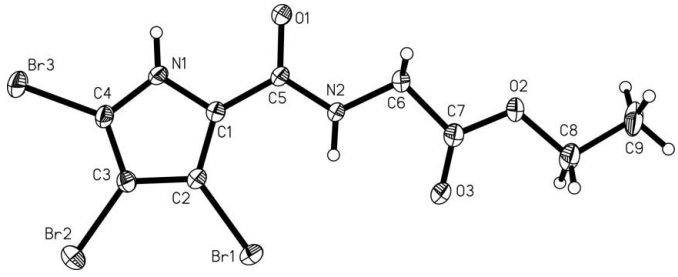

Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids for the non-H atoms are drawn at the 30% probability level.

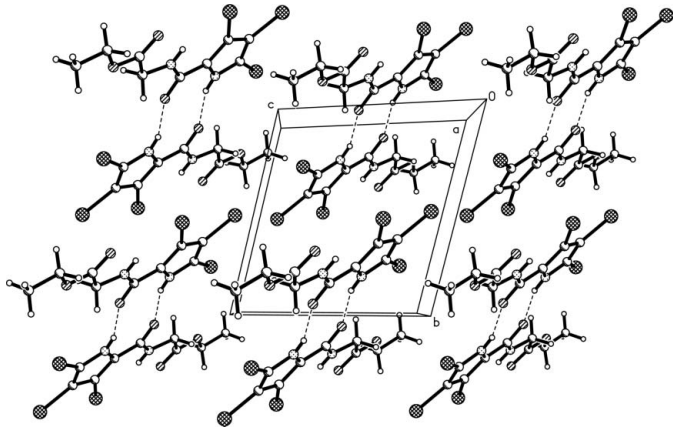

Figure 2
The packing of the title compound, viewed along the *a* axis, showing the centrosymmetric dimers formed by hydrogen bonds (dashed lines).

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