

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

D-Phenylglycinium bromide

Mohanadoss Parthasarathy,^a Kannan Arun Kumar^b and Rengasamy Gopalakrishnan^{a*}^aCrystal Research Laboratory, Department of Physics, Anna University, Chennai 600 025, India, and ^bDepartment of Chemistry, Loyola College (Autonomous), Chennai 600 034, India

Correspondence e-mail: krgkrishnan@annauniv.edu

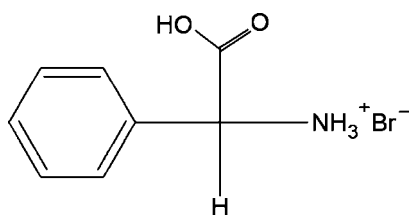
Received 30 January 2013; accepted 19 February 2013

Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.022; wR factor = 0.046; data-to-parameter ratio = 19.0.

In the crystal of the title salt, $\text{C}_8\text{H}_{10}\text{NO}_2^+\cdot\text{Br}^-$, the bromide anions and the phenylglycinium cations are linked through $\text{N}-\text{H}\cdots\text{Br}$, $\text{O}-\text{H}\cdots\text{Br}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, generating sheets lying parallel to (001).

Related literature

For a similar compound with a different halogen anion, see: Ravichandran *et al.* (1998). For related structures and background, see: Srinivasan *et al.* (2001); Bouchouit *et al.* (2004); Ramaswamy *et al.* (2001); Bouacida *et al.* (2006); Thomsen *et al.* (1994). For biological importance, see: Satyam *et al.* (1996); Jayasinghe *et al.* (1994); Chun *et al.* (2010); Thomas & West (2011).



Experimental

Crystal data

$\text{C}_8\text{H}_{10}\text{NO}_2^+\cdot\text{Br}^-$
 $M_r = 232.08$
 Orthorhombic, $P2_12_1$
 $a = 5.5240$ (5) Å
 $b = 7.4735$ (5) Å
 $c = 23.1229$ (18) Å

$V = 954.60$ (13) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 4.27$ mm⁻¹
 $T = 295$ K
 $0.35 \times 0.30 \times 0.25$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.317$, $T_{\max} = 0.415$

5824 measured reflections
 2170 independent reflections
 2003 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$
 $wR(F^2) = 0.046$
 $S = 1.03$
 2170 reflections
 114 parameters

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.29$ e Å⁻³
 Absolute structure: Flack (1983)
 Flack parameter: 0.011 (8)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1B}\cdots\text{Br1}^{\text{i}}$	0.89	2.54	3.3586 (17)	154
$\text{N1}-\text{H1C}\cdots\text{Br1}^{\text{ii}}$	0.89	2.57	3.429 (2)	163
$\text{N1}-\text{H1A}\cdots\text{Br1}$	0.89	2.45	3.3166 (18)	164
$\text{O1}-\text{H1D}\cdots\text{Br1}^{\text{iii}}$	0.82	2.39	3.2027 (17)	171
$\text{C7}-\text{H7}\cdots\text{O2}^{\text{iv}}$	0.98	2.59	3.527 (3)	159

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

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2 and SAINT (Bruker, 2004); data reduction: SAINT and XPREP (Bruker, 2004); program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: PLATON (Spek, 2009).

The authors are grateful to Professor K. Sivakumar, Department of Physics, Anna University, Chennai-25, for fruitful scientific discussions. The authors are thankful to the SAIF, IIT Madras, Chennai-36, India, for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2465).

References

- Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Bouacida, S., Merazig, H. & Benard-Rocherulle, P. (2006). *Acta Cryst.* **E62**, o838–o840.
- Bouchouit, K., Bendheif, L. & Benali-Cherif, N. (2004). *Acta Cryst.* **E60**, o272–o274.
- Bruker (2004). SADABS, APEX2, SAINT and XPREP. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chun, L. W., Yang, B. F., Hsiao, H. L., Tung, H. T., Ming, C. T. & Hui, P. W. (2010). *J. Biomed. Sci.* **17**, 71–78.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Jayasinghe, L. R., Datta, A., Ali, S. M., Zymunt, J., Van der Velde, D. G. & Georg, G. I. (1994). *J. Med. Chem.* **37**, 2981–2984.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Ramaswamy, S., Sridhar, B., Ramakrishnan, V. & Rajaram, R. K. (2001). *Acta Cryst.* **E57**, o1149–o1151.
- Ravichandran, S., Dattagupta, J. K. & Chakrabarti, C. (1998). *Acta Cryst.* **C54**, 499–501.
- Satyam, A., Hocker, M. D., Kanemaguire, K. A., Morgan, A. S., Villar, H. O. & Lyttle, M. H. (1996). *J. Med. Chem.* **39**, 1736–1747.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Srinivasan, N., Sridhar, B. & Rajaram, R. K. (2001). *Acta Cryst.* **E57**, o754–o756.
- Thomas, G. & West, G. B. (2011). *J. Pharm. Pharmacol.* **26**, 151–152.
- Thomsen, C., Boel, E. & Suzdak, P. D. (1994). *Eur. J. Pharmacol.* **267**, 77–84.

supporting information

Acta Cryst. (2013). E69, o470 [doi:10.1107/S1600536813004807]

D-Phenylglycinium bromide

Mohanadoss Parthasarathy, Kannan Arun Kumar and Rengasamy Gopalakrishnan

S1. Comment

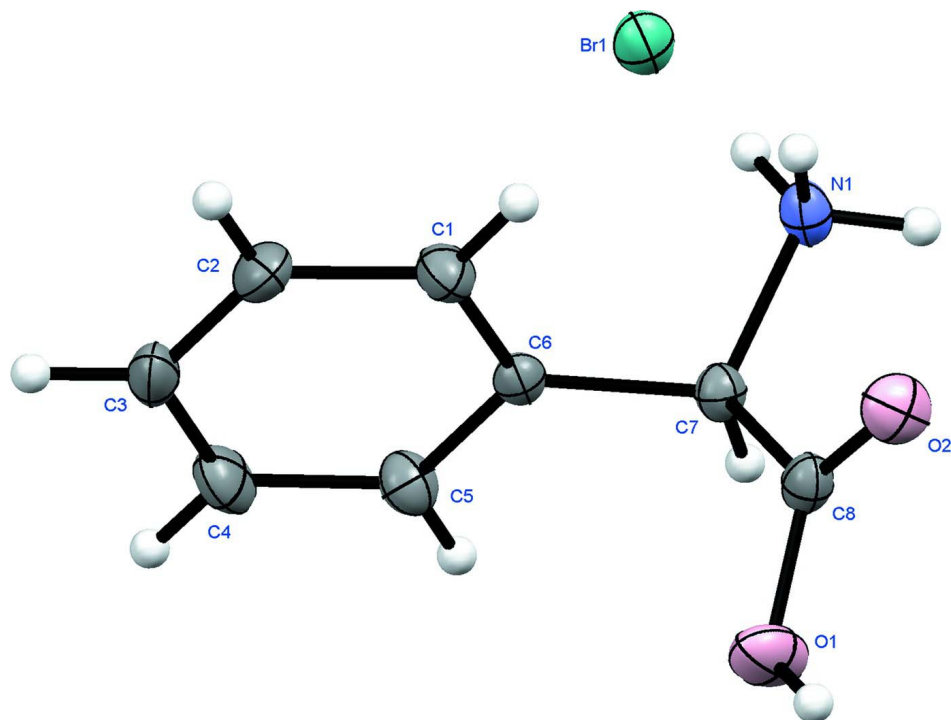
D-Phenylglycine is an important constituent in the production of semisynthetic penicillins and cephalosporins. Recently the usages of some phenylglycine derivatives in the synthesis of antitumor drugs and other pharmacological applications have been found to be increasing (Satyam et al., 1996; Jayasinghe et al., 1994). Phenylglycine has been reported as a delivery tool for improving l-dopa absorption (Chun et al., 2010) and also found to have anti-inflammatory activity (Thomas et al., 2011). The torsion angle N1-C7-C8-O1, which indicates the relative orientation of the carboxyl group and the amino N atom, is $15.5(3)^\circ$ and close to the corresponding value of $18.9(5)^\circ$ reported for D-Phenylglycine Hydrochloride (Ravichandran et al., 1998). The orientation of the phenyl ring as described by the torsion angle C5—C6—C7—N1 is $130.05(3)^\circ$. The intermolecular interaction between the molecular ions are primarily decided by hydrogen bonding. The hydrogen bonds N1—H1A \cdots Br1, N1—H1B \cdots Br1ⁱ [Symmetry code: (i) $-x+1, y-1/2, -z+3/2$], N1—H1C \cdots Br1ⁱⁱ [Symmetry code: (ii) $x-1, y, z$] and O1—H1D \cdots Br1ⁱⁱⁱ [Symmetry code: (iii) $x-1, y-1, z$] and C7—H7 \cdots O2^{iv} [Symmetry code: (iv) $x-1, y-1, z$] hydrogen bond interconnects the molecular ions to form an extensive two-dimensional molecular sheet parallel to (001) plane. Parallel stacking of these sheets along [0 0 1] direction constitute the molecular packing of the crystal.

S2. Experimental

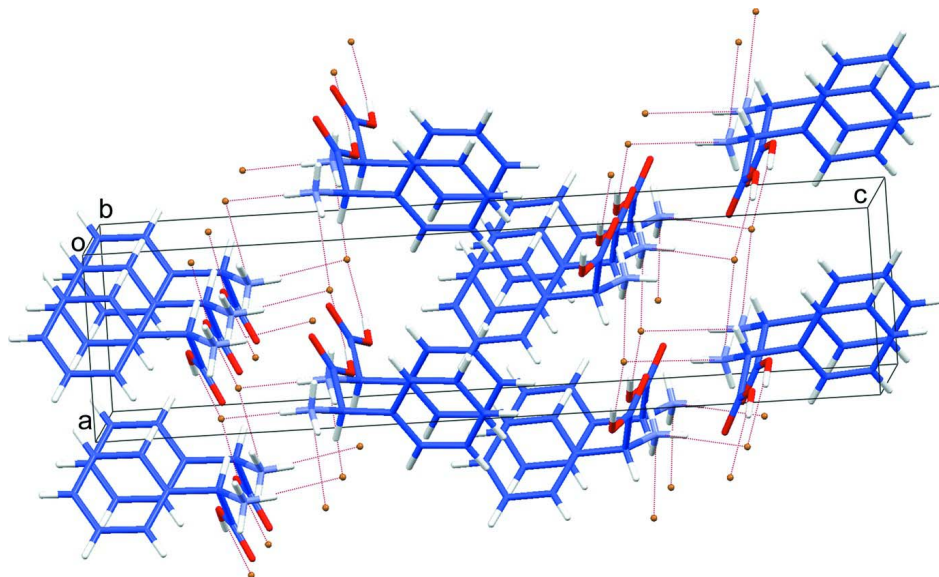
The title compound (I), was prepared by mixing a 1:1 ratio of *D*-Phenylglycine and hydrobromic acid in water solvent. The suitable single-crystal of the compound was selected for X-ray analysis from the above solution by slow evaporation method.

S3. Refinement

The hydrogen atoms associated with C atoms were identified from the difference electron density peaks and subsequently treated as riding atoms with distances of $d(\text{C-H}) = 0.98 \text{ \AA}$ (for CH) with $U_{\text{iso}}(\text{H}) = -1.5U_{\text{eq}}(\text{C})$ and $d(\text{C-H}) = 0.93 \text{ \AA}$ (for aromatic CH) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The carboxylic acid hydrogen was constrained to a distance of $d(\text{O-H}) = 0.82 \text{ \AA}$ with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ and the positions of NH_3 H atoms were also treated as riding about the parent atom.

**Figure 1**

Displacement ellipsoid plot of the molecular structure drawn at the 40% probability level.

**Figure 2**

Part of the crystal structure showing the two dimensional anionic-cationic (0 0 1) sheet formed through N—H \cdots Br, O—H \cdots Br and C—H \cdots O interactions viewed down *c* axis.

D-Phenylglycinium bromide*Crystal data*C₈H₁₀NO₂⁺·Br⁻ $M_r = 232.08$ Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

 $a = 5.5240$ (5) Å $b = 7.4735$ (5) Å $c = 23.1229$ (18) Å $V = 954.60$ (13) Å³ $Z = 4$ $F(000) = 464$ $D_x = 1.615$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3239 reflections

 $\theta = 2.6$ – 28.8° $\mu = 4.27$ mm⁻¹ $T = 295$ K

Block, colourless

 $0.35 \times 0.30 \times 0.25$ mm*Data collection*Bruker Kappa APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω and φ scanAbsorption correction: multi-scan
(*SADABS*; Bruker, 2004) $T_{\min} = 0.317$, $T_{\max} = 0.415$

5824 measured reflections

2170 independent reflections

2003 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.022$ $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.3^\circ$ $h = -6 \rightarrow 7$ $k = -9 \rightarrow 5$ $l = -28 \rightarrow 30$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.022$ $wR(F^2) = 0.046$ $S = 1.03$

2170 reflections

114 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2)]$ $(\Delta/\sigma)_{\max} = 0.003$ $\Delta\rho_{\max} = 0.25$ e Å⁻³ $\Delta\rho_{\min} = -0.29$ e Å⁻³Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0530 (13)

Absolute structure: Flack (1983)

Absolute structure parameter: 0.011 (8)

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1060 (5)	0.5444 (3)	0.56980 (9)	0.0370 (5)
H1	-0.0251	0.5884	0.5906	0.044*
C2	0.1209 (5)	0.5734 (3)	0.51081 (9)	0.0418 (6)

H2	-0.0006	0.6372	0.4921	0.050*
C3	0.3119 (5)	0.5092 (3)	0.47991 (9)	0.0424 (6)
H3	0.3196	0.5290	0.4402	0.051*
C4	0.4922 (5)	0.4160 (3)	0.50678 (9)	0.0443 (6)
H4	0.6229	0.3734	0.4855	0.053*
C5	0.4804 (4)	0.3849 (3)	0.56595 (9)	0.0355 (5)
H5	0.6024	0.3204	0.5842	0.043*
C6	0.2866 (4)	0.4500 (2)	0.59770 (7)	0.0264 (4)
C7	0.2695 (4)	0.4057 (2)	0.66152 (7)	0.0282 (5)
H7	0.4217	0.3499	0.6738	0.034*
C8	0.0650 (4)	0.2773 (3)	0.67328 (8)	0.0311 (5)
N1	0.2266 (4)	0.5687 (2)	0.69772 (6)	0.0345 (4)
H1A	0.3369	0.6514	0.6892	0.063 (8)*
H1B	0.2379	0.5399	0.7350	0.063 (8)*
H1C	0.0794	0.6117	0.6905	0.047 (7)*
O1	0.1199 (4)	0.1163 (2)	0.65347 (8)	0.0561 (5)
H1D	0.0051	0.0486	0.6586	0.084*
O2	-0.1206 (3)	0.3164 (2)	0.69632 (6)	0.0441 (4)
Br1	0.71415 (4)	0.82278 (3)	0.681529 (8)	0.03843 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0381 (14)	0.0388 (11)	0.0339 (11)	0.0062 (12)	0.0048 (10)	-0.0032 (10)
C2	0.0552 (17)	0.0342 (12)	0.0360 (12)	0.0066 (11)	-0.0030 (11)	0.0072 (10)
C3	0.0638 (18)	0.0364 (12)	0.0270 (10)	-0.0101 (13)	0.0087 (11)	0.0010 (9)
C4	0.0417 (16)	0.0496 (14)	0.0415 (14)	-0.0021 (12)	0.0170 (11)	-0.0082 (11)
C5	0.0277 (13)	0.0429 (12)	0.0360 (12)	0.0004 (11)	0.0035 (9)	-0.0027 (10)
C6	0.0261 (11)	0.0258 (9)	0.0273 (9)	-0.0062 (10)	0.0011 (9)	-0.0013 (7)
C7	0.0261 (13)	0.0316 (10)	0.0269 (9)	-0.0034 (10)	0.0012 (8)	0.0002 (7)
C8	0.0352 (14)	0.0323 (11)	0.0258 (11)	-0.0071 (10)	-0.0018 (9)	0.0014 (8)
N1	0.0377 (13)	0.0396 (9)	0.0262 (9)	-0.0137 (10)	0.0029 (8)	-0.0036 (7)
O1	0.0634 (15)	0.0333 (8)	0.0715 (12)	-0.0161 (9)	0.0238 (10)	-0.0057 (8)
O2	0.0328 (9)	0.0448 (9)	0.0547 (9)	-0.0106 (9)	0.0085 (7)	-0.0021 (8)
Br1	0.04061 (14)	0.03701 (12)	0.03766 (12)	-0.01269 (10)	0.00446 (10)	-0.00279 (9)

Geometric parameters (Å, °)

C1—C6	1.382 (3)	C6—C7	1.515 (2)
C1—C2	1.383 (3)	C7—N1	1.497 (2)
C1—H1	0.9300	C7—C8	1.507 (3)
C2—C3	1.361 (3)	C7—H7	0.9800
C2—H2	0.9300	C8—O2	1.192 (3)
C3—C4	1.365 (3)	C8—O1	1.323 (3)
C3—H3	0.9300	N1—H1A	0.8900
C4—C5	1.389 (3)	N1—H1B	0.8900
C4—H4	0.9300	N1—H1C	0.8900
C5—C6	1.386 (3)	O1—H1D	0.8200

C5—H5	0.9300		
C6—C1—C2	119.8 (2)	C5—C6—C7	119.15 (19)
C6—C1—H1	120.1	N1—C7—C8	107.37 (17)
C2—C1—H1	120.1	N1—C7—C6	112.12 (15)
C3—C2—C1	120.6 (2)	C8—C7—C6	111.19 (16)
C3—C2—H2	119.7	N1—C7—H7	108.7
C1—C2—H2	119.7	C8—C7—H7	108.7
C2—C3—C4	120.4 (2)	C6—C7—H7	108.7
C2—C3—H3	119.8	O2—C8—O1	125.1 (2)
C4—C3—H3	119.8	O2—C8—C7	124.73 (19)
C3—C4—C5	120.0 (2)	O1—C8—C7	110.16 (19)
C3—C4—H4	120.0	C7—N1—H1A	109.5
C5—C4—H4	120.0	C7—N1—H1B	109.5
C6—C5—C4	119.9 (2)	H1A—N1—H1B	109.5
C6—C5—H5	120.0	C7—N1—H1C	109.5
C4—C5—H5	120.0	H1A—N1—H1C	109.5
C1—C6—C5	119.30 (19)	H1B—N1—H1C	109.5
C1—C6—C7	121.4 (2)	C8—O1—H1D	109.5
C6—C1—C2—C3	0.2 (4)	C1—C6—C7—N1	-54.0 (3)
C1—C2—C3—C4	-0.3 (4)	C5—C6—C7—N1	130.1 (2)
C2—C3—C4—C5	0.5 (4)	C1—C6—C7—C8	66.2 (2)
C3—C4—C5—C6	-0.6 (3)	C5—C6—C7—C8	-109.7 (2)
C2—C1—C6—C5	-0.2 (3)	N1—C7—C8—O2	15.6 (3)
C2—C1—C6—C7	-176.2 (2)	C6—C7—C8—O2	-107.4 (2)
C4—C5—C6—C1	0.5 (3)	N1—C7—C8—O1	-165.48 (17)
C4—C5—C6—C7	176.5 (2)	C6—C7—C8—O1	71.5 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1B \cdots Br1 ⁱ	0.89	2.54	3.3586 (17)	154
N1—H1C \cdots Br1 ⁱⁱ	0.89	2.57	3.429 (2)	163
N1—H1A \cdots Br1	0.89	2.45	3.3166 (18)	164
O1—H1D \cdots Br1 ⁱⁱⁱ	0.82	2.39	3.2027 (17)	171
C7—H7 \cdots O2 ^{iv}	0.98	2.59	3.527 (3)	159

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$; (ii) $x-1, y, z$; (iii) $x-1, y-1, z$; (iv) $x+1, y, z$.