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Molecular and crystal structure of methyl 4-methyl-2,2-dioxo-1*H*-2λ⁶,1-benzothiazine-3-carboxylate

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Keywords: 1,2-benzothiazine derivatives; molecular and crystal structure; hydrogen bonding; π -stacking interaction.

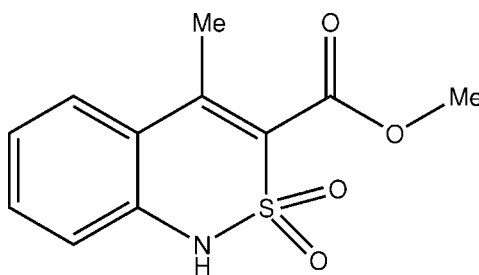
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The title compound, C₁₁H₁₁NO₄S, possesses weak analgesic properties and is a source compound for the synthesis of highly active analgesic and anti-inflammatory compounds. The benzothiazine ring adopts a conformation intermediate between twist-boat and sofa. The ester substituent is turned towards the endocyclic double bond because of steric repulsion. In the crystal, the molecules form columns along the [001] direction, bound by N—H···O hydrogen bonds and stacking interactions.

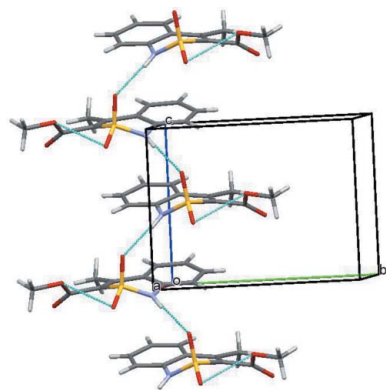
1. Chemical context

Methyl 4-methyl-2,2-dioxo-1*H*-2λ⁶,1-benzothiazine-3-carboxylate (**I**) displays moderate analgesic properties (Azotla-Cruz *et al.*, 2017) but has been used for the synthesis of highly active analgesic and anti-inflammatory compounds (Ukrainets *et al.*, 2018). Earlier it was shown (Ukrainets *et al.*, 2016*a,b*) that the biological properties of 2,1-benzothiazine derivatives depend to a considerable degree on their molecular and crystal structures. Thus knowledge of both the molecular and crystal structures of **I** is very important.



2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The dihydrothiazine heterocycle adopts a twist-boat conformation with puckering parameters (Zefirov *et al.*, 1990) $S = 0.57$, $\Theta = 53.3^\circ$, $\Psi = 25.2^\circ$. The S1 and C8 atoms deviate from the mean plane of the remaining ring atoms by 0.7941 (6) and 0.260 (2) Å, respectively. Some steric repulsion between the methyl substituent at the C7 atom and the ester group [the short intramolecular contact C11···O1 is 2.986 (5) Å compared to the van der Waals radii sum of 3.00 Å (Zefirov, 1997)] is compensated for by the formation of the intramolecular C11—H11C···O1 hydrogen bond (Table 1). As a



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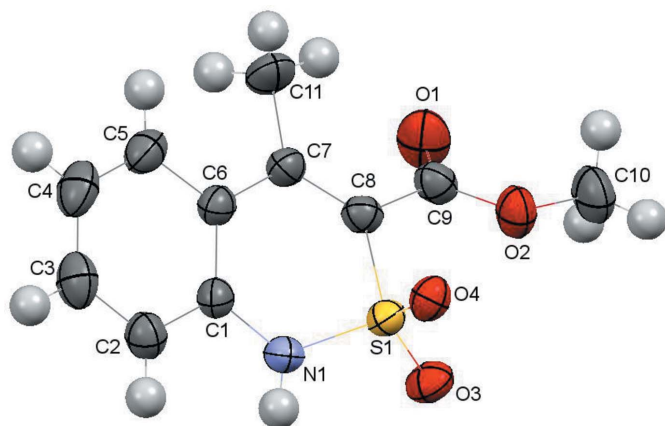


Figure 1
The molecular structure of **I** with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

result, the ester substituent is turned relative to the $C7=C8$ endocyclic double bond [$C7=C8-C9-O1$ torsion angle is $-35.2(5)^\circ$] and the $C7=C8$ [$1.359(4) \text{ \AA}$] and $C8-C9$ [$1.504(3) \text{ \AA}$] bonds are elongated compared to the standard values (Bürgi & Dunitz, 1994) of 1.326 and 1.455 \AA , respectively. The methyl group of the ester substituent is in an anti-periplanar conformation relative to the $C8-C9$ bond [$C8-C9-O2-C10 = 174.5(2)^\circ$].

3. Supramolecular features

In the crystal, molecules of **I** form columns along the $[001]$ direction (Fig. 2). Neighboring molecules within the column are linked by the $N1-H1N \cdots O4^i$ hydrogen bonds (Table 1)

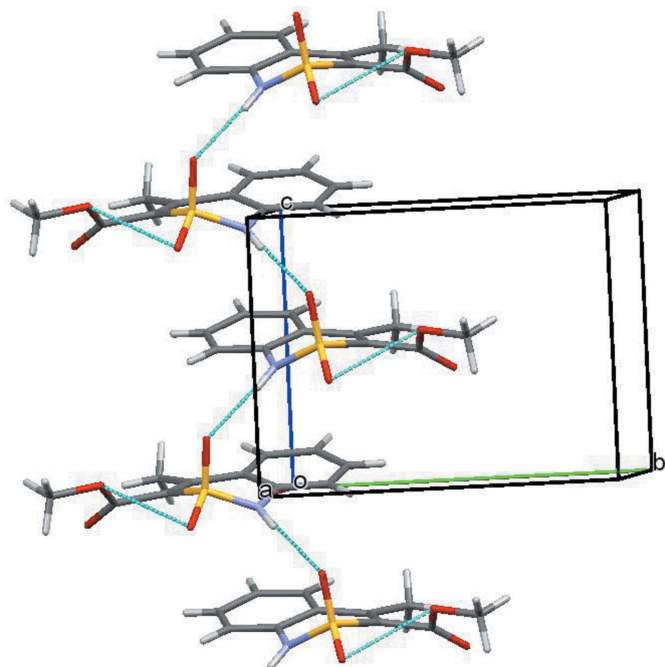


Figure 2
The packing showing columns of molecules along the c -axis direction.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C11-H11C \cdots O1$	0.96	2.24	2.986 (5)	133
$N1-H1N \cdots O4^i$	0.81 (4)	2.09 (4)	2.891 (3)	170 (4)
$C4-H4 \cdots O3^{ii}$	0.93	2.55	3.427 (3)	158

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

and π -stacking interactions with centroid-centroid distances of $3.870(2) \text{ \AA}$. The columns are connected by weak $C4-H4 \cdots O3^{ii}$ hydrogen bonds (Table 1).

4. Database survey

An search of the Cambridge Structural Database (Version 5.39, update February 2018; Groom *et al.*, 2016) revealed only three similar 1,2-benzothiazine derivatives with a methyl substituent at the $C7$ atom (VAZQEV and VAZQIZ, Azotla-Cruz *et al.*, 2017; OWUQII, Azotla-Cruz *et al.*, 2016). All of these compounds are substituted at the nitrogen atom and have very similar molecular structures. The structure VAZQEV differs from others by the *trans*-orientation of the

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{11}NO_4S$
M_r	253.27
Crystal system, space group	Monoclinic, Pc
Temperature (K)	293
a, b, c (\AA)	7.8367 (3), 9.6842 (4), 7.5006 (4)
β ($^\circ$)	93.468 (4)
V (\AA^3)	568.19 (4)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.29
Crystal size (mm)	$0.21 \times 0.18 \times 0.15$
Data collection	
Diffractometer	Agilent Xcalibur Sapphire3
Absorption correction	Multi-scan (CrysAlis RED; Agilent, 2012)
T_{\min}, T_{\max}	0.809, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5509, 3068, 2803
R_{int}	0.026
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.703
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.035, 0.085, 1.04
No. of reflections	3068
No. of parameters	160
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e \AA^{-3})	0.19, -0.21
Absolute structure	Flack x determined using 1127 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.04 (5)

Computer programs: CrysAlis CCD and CrysAlis RED (Agilent, 2012), SHELXS2014/7 (Sheldrick, 2008), SHELXL2014/7 (Sheldrick, 2015) and Mercury (Macrae *et al.*, 2008).

carbonyl group of the ester substituent relative to the endocyclic double bond.

4.1. Synthesis and crystallization

Methyl (chlorosulfonyl)acetate (1.90 g, 0.011 mol) was added dropwise with stirring to a solution of *ortho*-aminoacetophenone (1.35 g, 0.010 mol) and triethylamine (1.54 mL, 0.011 mol) in CH₂Cl₂ (20 mL) and cooled to 268–273 K. After 10 h, water (50 mL) was added to the reaction mixture, which was then acidified to pH 4 with 1 N HCl and mixed thoroughly. The organic layer was separated off, dried over anhydrous CaCl₂, and the solvent distilled (at reduced pressure at the end). The resulting anilide was subjected to heterocyclization without purification. A solution of sodium methylate in anhydrous methanol [from metallic sodium (0.69 g, 0.030 mol) and absolute methanol (15 mL)], the mixture was boiled and then kept for 15 h at room temperature. The reaction mixture was diluted with cold water and acidified with 1 N HCl to pH 4. Finally, the solid ester, **I**, was separated by filtration, washed with water, and dried in air giving colourless block-shaped crystals, yield: 2.25 g (89%); m.p. 476–578 K (methanol); $R_f = 0.37$. ¹H NMR (400 MHz, DMSO-*d*₆): δ 11.84 (*br s*, 1H, NH), 7.79 (*d*, 1H, $J = 7.6$ Hz, H-5), 7.49 (*t*, 1H, $J = 7.2$ Hz, H-7), 7.22 (*t*, 1H, $J = 7.6$ Hz, H-6), 7.12 (*d*, 1H, $J = 8.0$ Hz, H-8), 3.84 (*s*, 3H, OCH₃), 2.46 (*s*, 3H, 4-CH₃, coincides with the signal of residual protons DMSO-*d*₆). ¹³C-NMR (100 MHz, DMSO-*d*₆ + CDCl₃): δ 161.6 (C=O), 147.7, 138.2, 132.2, 127.4, 127.1, 123.0, 121.3, 118.8, 52.9 (OCH₃), 17.5 (4-CH₃). MS (m/z , %): 253 [M]⁺ (4.4), 252 [$M - H$]⁺ (1.5), 221 [$M - CH_3OH$]⁺ (8.4), 195 (80.2), 119 (75.3), 103 (17.0), 93 (100), 92 (59.5), 77 (50.0). Analysis calculated for C₁₁H₁₁NO₄S: C, 52.16; H, 4.38; N, 5.53; S 12.66%. Found: C, 52.07; H, 4.30; N, 5.46; S 12.72%.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All of the H atoms were located in difference-Fourier maps. The N-bound H atoms were refined isotropically. The C-bound H atoms were included in calculated positions and treated as riding: C–H = 0.96 Å with $U_{iso}(H) = 1.5U_{eq}(C)$ for the methyl groups and C–H = 0.93 Å with $U_{iso}(H) = 1.2U_{eq}(C)$ for all others.

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Computing details

Data collection: *CrysAlis CCD* (Agilent, 2012); cell refinement: *CrysAlis RED* (Agilent, 2012); data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: *SHELXS2014/7* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014/7* (Sheldrick, 2015).

Methyl 4-methyl-2,2-dioxo-1*H*-2λ⁶,1-benzothiazine-3-carboxylate

Crystal data

C₁₁H₁₁NO₄S

M_r = 253.27

Monoclinic, *Pc*

a = 7.8367 (3) Å

b = 9.6842 (4) Å

c = 7.5006 (4) Å

β = 93.468 (4)°

V = 568.19 (4) Å³

Z = 2

F(000) = 264

D_x = 1.480 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 2089 reflections

θ = 4.2–30.6°

μ = 0.29 mm⁻¹

T = 293 K

Block, colourless

0.21 × 0.18 × 0.15 mm

Data collection

Agilent Xcalibur Sapphire3
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Detector resolution: 16.1827 pixels mm⁻¹

ω-scans

Absorption correction: multi-scan
(*CrysAlis RED*; Agilent, 2012)

T_{min} = 0.809, *T_{max}* = 1.000

5509 measured reflections

3068 independent reflections

2803 reflections with *I* > 2σ(*I*)

R_{int} = 0.026

θ_{max} = 30.0°, θ_{min} = 3.4°

h = -10→11

k = -9→13

l = -10→10

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.035

wR(*F*²) = 0.085

S = 1.04

3068 reflections

160 parameters

2 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent
and constrained refinement

w = 1/[σ²(*F_o*²) + (0.0399*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.19 e Å⁻³

Δρ_{min} = -0.21 e Å⁻³

Absolute structure: Flack x determined using
1127 quotients $[(F^+)-(F^-)]/[(F^+)+(F^-)]$ (Parsons et
al., 2013)
Absolute structure parameter: 0.04 (5)

Special details

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.82278 (8)	0.16925 (5)	0.52933 (8)	0.03401 (14)
O1	0.6143 (4)	0.5195 (2)	0.4202 (4)	0.0679 (7)
O2	0.8581 (3)	0.4555 (2)	0.5668 (3)	0.0514 (5)
O3	0.9468 (3)	0.2019 (2)	0.4041 (3)	0.0477 (5)
O4	0.8846 (2)	0.15240 (18)	0.7126 (2)	0.0416 (4)
N1	0.7250 (3)	0.0322 (2)	0.4558 (3)	0.0420 (5)
H1N	0.780 (5)	-0.012 (4)	0.388 (5)	0.064 (11)*
C1	0.5686 (3)	-0.0083 (2)	0.5189 (3)	0.0364 (5)
C2	0.5276 (4)	-0.1480 (3)	0.5226 (4)	0.0459 (6)
H2	0.6049	-0.2136	0.4864	0.055*
C3	0.3720 (5)	-0.1882 (3)	0.5802 (4)	0.0558 (8)
H3	0.3433	-0.2814	0.5814	0.067*
C4	0.2584 (4)	-0.0915 (4)	0.6361 (4)	0.0570 (8)
H4	0.1550	-0.1198	0.6786	0.068*
C5	0.2967 (4)	0.0469 (3)	0.6297 (4)	0.0469 (6)
H5	0.2174	0.1112	0.6652	0.056*
C6	0.4538 (3)	0.0927 (3)	0.5705 (3)	0.0374 (5)
C7	0.4909 (3)	0.2408 (3)	0.5508 (3)	0.0384 (5)
C8	0.6512 (3)	0.2858 (3)	0.5222 (3)	0.0365 (5)
C9	0.7020 (4)	0.4337 (3)	0.4961 (4)	0.0431 (6)
C10	0.9327 (5)	0.5898 (3)	0.5392 (5)	0.0634 (9)
H10A	1.0462	0.5924	0.5949	0.095*
H10B	0.8640	0.6595	0.5908	0.095*
H10C	0.9375	0.6066	0.4134	0.095*
C11	0.3470 (5)	0.3403 (3)	0.5685 (6)	0.0599 (10)
H11A	0.3055	0.3331	0.6860	0.090*
H11B	0.2562	0.3192	0.4811	0.090*
H11C	0.3869	0.4326	0.5498	0.090*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0300 (3)	0.0345 (2)	0.0379 (3)	0.0013 (2)	0.0052 (2)	0.0017 (2)
O1	0.0679 (16)	0.0430 (11)	0.0907 (18)	0.0062 (10)	-0.0134 (14)	0.0153 (11)
O2	0.0499 (13)	0.0386 (9)	0.0646 (14)	-0.0088 (9)	-0.0041 (10)	0.0034 (8)

O3	0.0423 (11)	0.0504 (11)	0.0519 (12)	0.0000 (9)	0.0161 (9)	0.0048 (9)
O4	0.0372 (10)	0.0453 (10)	0.0421 (11)	-0.0009 (7)	-0.0014 (8)	0.0061 (7)
N1	0.0383 (12)	0.0385 (11)	0.0505 (13)	-0.0010 (9)	0.0116 (10)	-0.0106 (9)
C1	0.0333 (12)	0.0388 (12)	0.0367 (13)	-0.0032 (10)	0.0000 (10)	-0.0034 (9)
C2	0.0472 (16)	0.0391 (13)	0.0508 (16)	-0.0035 (11)	-0.0007 (13)	0.0006 (11)
C3	0.059 (2)	0.0488 (16)	0.059 (2)	-0.0186 (14)	0.0022 (15)	0.0046 (12)
C4	0.0461 (17)	0.072 (2)	0.0530 (17)	-0.0189 (15)	0.0062 (13)	0.0059 (15)
C5	0.0349 (13)	0.0617 (17)	0.0444 (15)	-0.0021 (12)	0.0040 (12)	-0.0013 (12)
C6	0.0323 (12)	0.0426 (13)	0.0369 (12)	0.0005 (10)	-0.0004 (9)	-0.0029 (9)
C7	0.0328 (12)	0.0405 (13)	0.0416 (13)	0.0036 (10)	-0.0010 (10)	-0.0047 (10)
C8	0.0370 (13)	0.0343 (11)	0.0383 (13)	0.0052 (9)	0.0010 (10)	-0.0012 (9)
C9	0.0474 (15)	0.0347 (12)	0.0469 (16)	0.0045 (11)	-0.0004 (13)	-0.0027 (10)
C10	0.069 (2)	0.0425 (17)	0.078 (2)	-0.0173 (15)	0.0017 (18)	-0.0029 (15)
C11	0.0372 (17)	0.0518 (15)	0.091 (3)	0.0120 (13)	0.0055 (18)	-0.0072 (15)

Geometric parameters (Å, °)

S1—O3	1.4271 (19)	C1—C6	1.400 (3)
S1—O4	1.4391 (19)	C2—C3	1.375 (5)
S1—N1	1.613 (2)	C3—C4	1.375 (5)
S1—C8	1.754 (3)	C4—C5	1.375 (4)
O1—C9	1.199 (4)	C5—C6	1.406 (4)
O2—C9	1.321 (4)	C6—C7	1.472 (4)
O2—C10	1.446 (4)	C7—C8	1.359 (4)
N1—C1	1.397 (3)	C7—C11	1.496 (4)
C1—C2	1.391 (3)	C8—C9	1.503 (4)
O3—S1—O4	116.79 (13)	C4—C5—C6	121.1 (3)
O3—S1—N1	106.58 (13)	C1—C6—C5	117.2 (2)
O4—S1—N1	111.04 (12)	C1—C6—C7	121.3 (2)
O3—S1—C8	112.85 (12)	C5—C6—C7	121.4 (3)
O4—S1—C8	108.38 (12)	C8—C7—C6	121.2 (2)
N1—S1—C8	99.88 (13)	C8—C7—C11	121.1 (3)
C9—O2—C10	117.3 (2)	C6—C7—C11	117.7 (2)
C1—N1—S1	121.58 (18)	C7—C8—C9	125.5 (2)
C2—C1—N1	119.2 (2)	C7—C8—S1	120.2 (2)
C2—C1—C6	121.4 (2)	C9—C8—S1	114.1 (2)
N1—C1—C6	119.3 (2)	O1—C9—O2	124.7 (3)
C3—C2—C1	119.5 (3)	O1—C9—C8	125.0 (3)
C2—C3—C4	120.4 (3)	O2—C9—C8	110.3 (2)
C5—C4—C3	120.5 (3)		
O3—S1—N1—C1	-163.3 (2)	C1—C6—C7—C11	166.1 (3)
O4—S1—N1—C1	68.5 (2)	C5—C6—C7—C11	-9.2 (4)
C8—S1—N1—C1	-45.7 (2)	C6—C7—C8—C9	178.5 (2)
S1—N1—C1—C2	-149.9 (2)	C11—C7—C8—C9	-3.3 (4)
S1—N1—C1—C6	32.6 (3)	C6—C7—C8—S1	-6.1 (3)
N1—C1—C2—C3	-178.3 (3)	C11—C7—C8—S1	172.1 (2)

C6—C1—C2—C3	-0.8 (4)	O3—S1—C8—C7	145.0 (2)
C1—C2—C3—C4	-0.9 (5)	O4—S1—C8—C7	-84.1 (2)
C2—C3—C4—C5	2.1 (5)	N1—S1—C8—C7	32.2 (2)
C3—C4—C5—C6	-1.6 (4)	O3—S1—C8—C9	-39.2 (2)
C2—C1—C6—C5	1.3 (4)	O4—S1—C8—C9	91.8 (2)
N1—C1—C6—C5	178.8 (2)	N1—S1—C8—C9	-151.97 (19)
C2—C1—C6—C7	-174.3 (2)	C10—O2—C9—O1	-4.8 (5)
N1—C1—C6—C7	3.2 (4)	C10—O2—C9—C8	174.5 (2)
C4—C5—C6—C1	-0.1 (4)	C7—C8—C9—O1	-35.2 (5)
C4—C5—C6—C7	175.5 (2)	S1—C8—C9—O1	149.2 (3)
C1—C6—C7—C8	-15.6 (4)	C7—C8—C9—O2	145.5 (3)
C5—C6—C7—C8	169.1 (3)	S1—C8—C9—O2	-30.1 (3)

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C11—H11C...O1	0.96	2.24	2.986 (5)	133
N1—H1N...O4 ⁱ	0.81 (4)	2.09 (4)	2.891 (3)	170 (4)
C4—H4...O3 ⁱⁱ	0.93	2.55	3.427 (3)	158

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