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2-(4-Bromophenyl)-2-oxoethyl naphthalene-1-carboxylate

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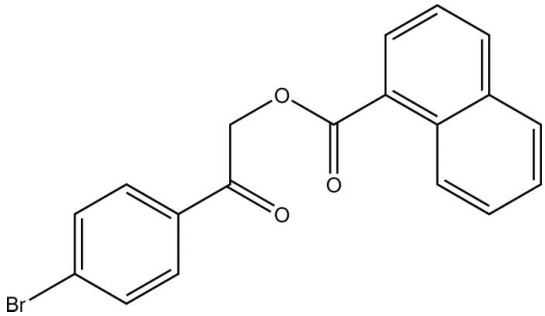
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.080; data-to-parameter ratio = 18.5.

In the title compound, $C_{19}H_{13}BrO_3$, an ester of 1-naphthoic acid with an aromatic alcohol, the least-squares planes defined by the C atoms of the respective aromatic systems enclose an angle of 77.20 (5)°. In the crystal, C—H···O contacts connect the molecules into undulating sheets parallel (100).

Related literature

For general information about phenacyl bromide derivatives, see: Rather & Reid (1919). For the photolytic properties of phenyl benzoates, see: Sheehan & Umezaw (1973); Ruzicka *et al.* (2002); Litera *et al.* (2006). For synthetic applications of phenyl benzoates, see: Huang *et al.* (1996); Gandhi *et al.* (1995). For graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{19}H_{13}BrO_3$
M_r = 369.20
Monoclinic, $P2_1/c$
a = 5.2888 (2) Å
b = 14.9139 (6) Å
c = 20.0275 (8) Å
β = 100.867 (2)°
V = 1551.37 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.66 mm^{-1}
T = 200 K
0.35 × 0.16 × 0.07 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2010)
T_{min} = 0.656, T_{max} = 0.746
14937 measured reflections
3848 independent reflections
2712 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.080
S = 1.02
3848 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O1^{i}	0.95	2.41	3.240 (3)	146
C23—H23···O3^{ii}	0.95	2.51	3.292 (3)	140

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

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QK2054).

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supporting information

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S1. Comment

For decades, phenacyl bromide derivatives have found ample application in the identification of organic acids (Rather & Reid, 1919). These compounds can be photolysed under neutral and mild conditions (Sheehan & Umezaw, 1973; Ruzicka *et al.*, 2002; Litera *et al.*, 2006). They also find application in the field of synthetic chemistry such as in the synthesis of oxazoles and imidazoles (Huang *et al.*, 1996) as well as benzoxazepine (Gandhi *et al.*, 1995). In continuation of our research focused on the crystal structures of medical compounds, the title compound was synthesized.

The planes defined by the atoms of the carboxy group on the one hand and the non-hydrogen atoms of the CH_2–C═O moiety intersect at an angle of 78.8 (3) °. The least-squares planes defined by the carbon atoms of the respective aromatic systems enclose an angle of 77.20 (5) ° (Fig. 1).

In the crystal, intermolecular C–H···O contacts are observed whose range falls by more than 0.2 Å below the sum of van-der-Waals radii of the atoms participating. One of the hydrogen atoms of the brominated phenyl moiety and the oxygen atom of the keto group give rise to centrosymmetric dimers. Additionally, the hydrogen atom in *para* position to the carboxy moiety forms a C–H···O contact to the double bonded oxygen atom of exactly this group in a neighbouring molecule. In total, the molecules are connected to undulated sheets parallel (100). In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the descriptor for these contacts is $C^1_1(7)R^2_2(10)$ on the unary level. Information about metrical parameters as well as the symmetry of those contacts has been summarized in Table 1. The shortest intercentroid distance between two aromatic systems was measured at 4.7943 (14) Å and is apparent between the two different rings in the naphthoic acid moiety and its symmetry-generated equivalents (Fig. 2).

S2. Experimental

A mixture of naphthalene-1-carboxylic acid (0.1 g, 0.5 mmol), potassium carbonate (0.087 g, 0.63 mmol) and 2-bromo-1-(4-bromophenyl)ethanone (0.174 g, 0.63 mmol) in dimethylformamide (5 ml) was stirred at room temperature for 2 h. After completion of the reaction, the reaction mixture was poured into ice-cold water. The solid product obtained was filtered, washed with water and recrystallized from ethanol (yield: 0.19 g, 90.4%).

S3. Refinement

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms and C–H 0.99 Å for the methylene group) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C).

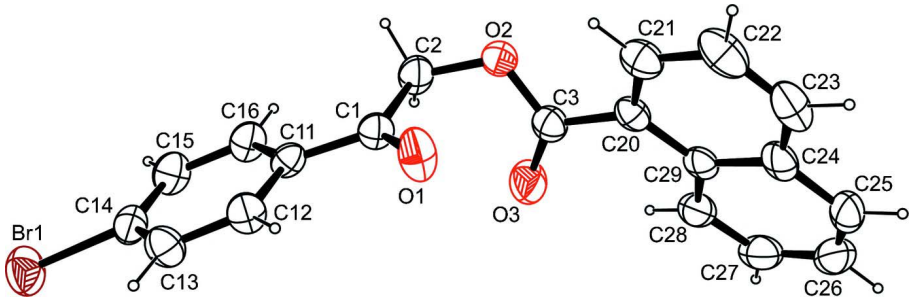

Figure 1

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level).

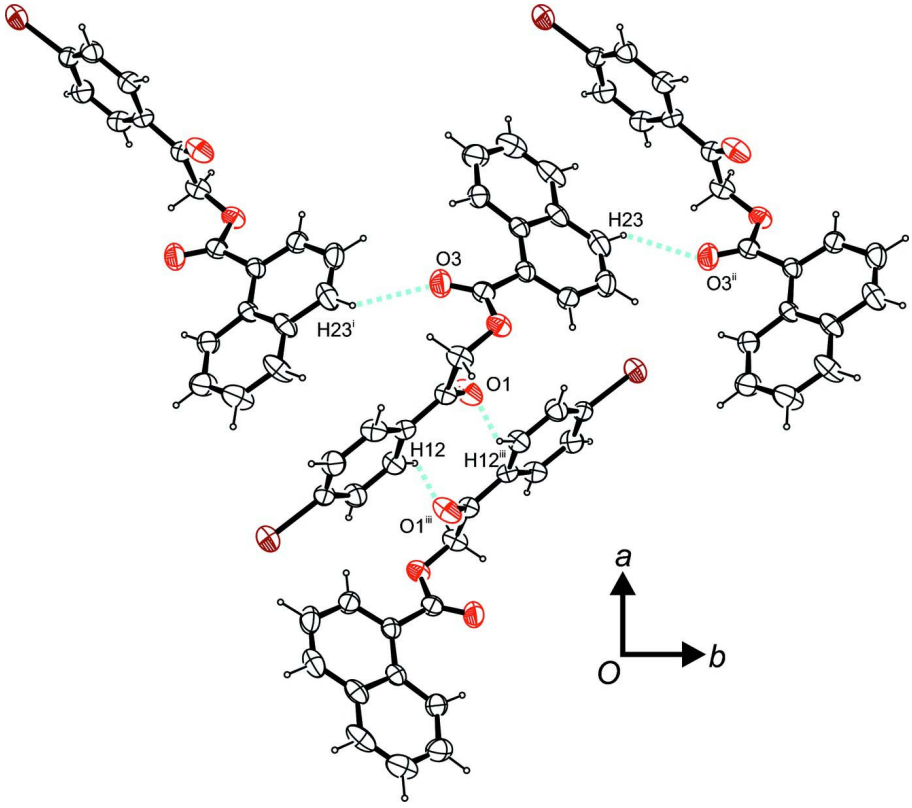

Figure 2

Intermolecular contacts, viewed along [-1 0 0]. Symmetry operators: [i] -*x* + 1, *y* - 1/2, -*z* + 1/2; [ii] -*x* + 1, *y* + 1/2, -*z* + 1/2; [iii] -*x* + 1, -*y*, -*z*.

2-(4-Bromophenyl)-2-oxoethyl naphthalene-1-carboxylate

Crystal data

$C_{19}H_{13}BrO_3$
M_r = 369.20
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 5.2888 (2) Å
b = 14.9139 (6) Å
c = 20.0275 (8) Å
β = 100.867 (2)°
V = 1551.37 (11) Å^3
Z = 4
F(000) = 744
D_x = 1.581 Mg m^{-3}

Melting point = 406–404 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4237 reflections
θ = 2.5–24.2°
μ = 2.66 mm^{-1}
T = 200 K
Platelet, colourless
0.35 × 0.16 × 0.07 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2010)
T_{min} = 0.656, T_{max} = 0.746
14937 measured reflections
3848 independent reflections
2712 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.4°, θ_{min} = 1.7°
h = −6→7
k = −19→19
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.080
S = 1.02
3848 reflections
208 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 0.6176P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Br1	1.05155 (5)	−0.337459 (17)	−0.082915 (14)	0.05483 (11)
O1	0.7161 (3)	0.01926 (12)	0.09358 (8)	0.0504 (4)
O2	1.0379 (3)	0.07451 (10)	0.20022 (7)	0.0383 (3)
O3	0.8211 (3)	−0.03018 (11)	0.24600 (9)	0.0561 (5)
C1	0.9200 (4)	−0.02042 (15)	0.10258 (10)	0.0324 (5)
C2	1.1300 (4)	0.00494 (16)	0.16236 (11)	0.0392 (5)
H2A	1.1784	−0.0480	0.1918	0.047*
H2B	1.2847	0.0257	0.1457	0.047*
C3	0.8587 (4)	0.04763 (15)	0.23606 (11)	0.0356 (5)
C11	0.9659 (4)	−0.09457 (14)	0.05693 (10)	0.0301 (4)
C12	0.7748 (4)	−0.11433 (15)	0.00128 (11)	0.0364 (5)
H12	0.6242	−0.0782	−0.0078	0.044*
C13	0.7996 (4)	−0.18520 (15)	−0.04088 (12)	0.0396 (5)
H13	0.6679	−0.1981	−0.0789	0.047*
C14	1.0190 (4)	−0.23729 (14)	−0.02702 (11)	0.0359 (5)
C15	1.2131 (4)	−0.21891 (16)	0.02701 (12)	0.0411 (5)
H15	1.3637	−0.2550	0.0356	0.049*
C16	1.1865 (4)	−0.14699 (15)	0.06894 (11)	0.0376 (5)
H16	1.3206	−0.1335	0.1063	0.045*
C20	0.7119 (4)	0.12388 (14)	0.25724 (10)	0.0317 (4)
C21	0.6958 (4)	0.20194 (15)	0.22036 (11)	0.0401 (5)

H21	0.7954	0.2086	0.1858	0.048*
C22	0.5341 (5)	0.27215 (16)	0.23293 (13)	0.0505 (6)
H22	0.5277	0.3263	0.2077	0.061*
C23	0.3868 (5)	0.26250 (17)	0.28116 (13)	0.0486 (6)
H23	0.2727	0.3093	0.2880	0.058*
C24	0.3998 (4)	0.18466 (16)	0.32119 (12)	0.0397 (5)
C25	0.2488 (4)	0.17481 (19)	0.37226 (14)	0.0517 (7)
H25	0.1312	0.2208	0.3785	0.062*
C26	0.2694 (5)	0.1008 (2)	0.41240 (14)	0.0572 (7)
H26	0.1656	0.0950	0.4460	0.069*
C27	0.4432 (5)	0.03358 (19)	0.40408 (13)	0.0525 (7)
H27	0.4620	−0.0169	0.4335	0.063*
C28	0.5868 (4)	0.03887 (16)	0.35440 (11)	0.0406 (5)
H28	0.7011	−0.0086	0.3491	0.049*
C29	0.5685 (4)	0.11383 (14)	0.31069 (10)	0.0316 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0741 (2)	0.04066 (15)	0.05347 (17)	0.00681 (13)	0.02159 (13)	−0.00796 (12)
O1	0.0365 (9)	0.0641 (12)	0.0454 (10)	0.0178 (8)	−0.0056 (7)	−0.0172 (8)
O2	0.0327 (8)	0.0439 (9)	0.0381 (8)	−0.0067 (7)	0.0063 (6)	−0.0090 (7)
O3	0.0770 (12)	0.0334 (10)	0.0665 (12)	0.0033 (9)	0.0357 (10)	−0.0005 (8)
C1	0.0258 (10)	0.0375 (12)	0.0325 (11)	0.0027 (9)	0.0020 (8)	−0.0001 (9)
C2	0.0280 (11)	0.0523 (14)	0.0365 (12)	0.0004 (10)	0.0040 (9)	−0.0084 (10)
C3	0.0354 (11)	0.0387 (13)	0.0314 (11)	−0.0017 (9)	0.0029 (9)	−0.0038 (9)
C11	0.0241 (10)	0.0378 (12)	0.0280 (10)	0.0017 (8)	0.0045 (8)	0.0034 (9)
C12	0.0293 (11)	0.0410 (13)	0.0367 (12)	0.0059 (9)	0.0007 (9)	−0.0017 (9)
C13	0.0378 (12)	0.0413 (13)	0.0365 (12)	0.0011 (10)	−0.0009 (9)	−0.0036 (10)
C14	0.0439 (12)	0.0322 (12)	0.0349 (12)	0.0037 (10)	0.0156 (10)	0.0017 (9)
C15	0.0343 (11)	0.0449 (14)	0.0443 (13)	0.0134 (10)	0.0083 (10)	0.0025 (10)
C16	0.0258 (10)	0.0519 (15)	0.0333 (11)	0.0064 (9)	0.0009 (9)	0.0000 (10)
C20	0.0306 (10)	0.0303 (11)	0.0311 (11)	−0.0012 (9)	−0.0020 (8)	−0.0042 (9)
C21	0.0464 (13)	0.0365 (12)	0.0344 (12)	−0.0041 (10)	0.0002 (10)	0.0003 (10)
C22	0.0635 (16)	0.0312 (13)	0.0491 (15)	0.0058 (12)	−0.0093 (13)	0.0008 (11)
C23	0.0470 (14)	0.0399 (14)	0.0519 (15)	0.0100 (11)	−0.0082 (12)	−0.0152 (11)
C24	0.0323 (11)	0.0432 (13)	0.0390 (12)	−0.0018 (10)	−0.0046 (9)	−0.0175 (10)
C25	0.0337 (12)	0.0657 (19)	0.0556 (16)	−0.0054 (12)	0.0080 (11)	−0.0334 (14)
C26	0.0491 (15)	0.076 (2)	0.0510 (16)	−0.0235 (14)	0.0201 (12)	−0.0227 (14)
C27	0.0607 (16)	0.0560 (17)	0.0428 (14)	−0.0237 (13)	0.0147 (12)	−0.0050 (12)
C28	0.0440 (12)	0.0374 (13)	0.0404 (13)	−0.0073 (10)	0.0078 (10)	−0.0025 (10)
C29	0.0279 (10)	0.0334 (12)	0.0302 (11)	−0.0044 (9)	−0.0025 (8)	−0.0079 (8)

Geometric parameters (Å, º)

Br1—C14	1.894 (2)	C16—H16	0.9500
O1—C1	1.213 (2)	C20—C21	1.373 (3)
O2—C3	1.353 (3)	C20—C29	1.432 (3)

O2—C2	1.424 (2)	C21—C22	1.404 (3)
O3—C3	1.200 (3)	C21—H21	0.9500
C1—C11	1.484 (3)	C22—C23	1.358 (4)
C1—C2	1.520 (3)	C22—H22	0.9500
C2—H2A	0.9900	C23—C24	1.405 (3)
C2—H2B	0.9900	C23—H23	0.9500
C3—C20	1.483 (3)	C24—C25	1.419 (3)
C11—C16	1.387 (3)	C24—C29	1.424 (3)
C11—C12	1.388 (3)	C25—C26	1.357 (4)
C12—C13	1.374 (3)	C25—H25	0.9500
C12—H12	0.9500	C26—C27	1.392 (4)
C13—C14	1.380 (3)	C26—H26	0.9500
C13—H13	0.9500	C27—C28	1.363 (3)
C14—C15	1.371 (3)	C27—H27	0.9500
C15—C16	1.386 (3)	C28—C29	1.412 (3)
C15—H15	0.9500	C28—H28	0.9500

C3—O2—C2	114.25 (17)	C21—C20—C29	120.2 (2)
O1—C1—C11	121.08 (18)	C21—C20—C3	118.6 (2)
O1—C1—C2	119.53 (19)	C29—C20—C3	120.87 (19)
C11—C1—C2	119.38 (17)	C20—C21—C22	120.9 (2)
O2—C2—C1	109.17 (16)	C20—C21—H21	119.5
O2—C2—H2A	109.8	C22—C21—H21	119.5
C1—C2—H2A	109.8	C23—C22—C21	119.9 (2)
O2—C2—H2B	109.8	C23—C22—H22	120.0
C1—C2—H2B	109.8	C21—C22—H22	120.0
H2A—C2—H2B	108.3	C22—C23—C24	121.4 (2)
O3—C3—O2	121.9 (2)	C22—C23—H23	119.3
O3—C3—C20	125.6 (2)	C24—C23—H23	119.3
O2—C3—C20	112.42 (19)	C23—C24—C25	121.6 (2)
C16—C11—C12	118.8 (2)	C23—C24—C29	119.6 (2)
C16—C11—C1	122.91 (18)	C25—C24—C29	118.9 (2)
C12—C11—C1	118.29 (17)	C26—C25—C24	121.2 (2)
C13—C12—C11	121.14 (19)	C26—C25—H25	119.4
C13—C12—H12	119.4	C24—C25—H25	119.4
C11—C12—H12	119.4	C25—C26—C27	119.7 (2)
C12—C13—C14	118.9 (2)	C25—C26—H26	120.1
C12—C13—H13	120.6	C27—C26—H26	120.1
C14—C13—H13	120.6	C28—C27—C26	121.1 (3)
C15—C14—C13	121.5 (2)	C28—C27—H27	119.4
C15—C14—Br1	119.07 (16)	C26—C27—H27	119.4
C13—C14—Br1	119.46 (17)	C27—C28—C29	121.0 (2)
C14—C15—C16	119.11 (19)	C27—C28—H28	119.5
C14—C15—H15	120.4	C29—C28—H28	119.5
C16—C15—H15	120.4	C28—C29—C24	117.9 (2)
C15—C16—C11	120.6 (2)	C28—C29—C20	124.22 (19)
C15—C16—H16	119.7	C24—C29—C20	117.9 (2)
C11—C16—H16	119.7		

C3—O2—C2—C1	71.6 (2)	O2—C3—C20—C29	−162.04 (17)
O1—C1—C2—O2	1.4 (3)	C29—C20—C21—C22	−1.7 (3)
C11—C1—C2—O2	−177.41 (18)	C3—C20—C21—C22	172.2 (2)
C2—O2—C3—O3	14.5 (3)	C20—C21—C22—C23	−1.4 (3)
C2—O2—C3—C20	−162.58 (16)	C21—C22—C23—C24	2.6 (4)
O1—C1—C11—C16	−173.9 (2)	C22—C23—C24—C25	179.1 (2)
C2—C1—C11—C16	4.9 (3)	C22—C23—C24—C29	−0.7 (3)
O1—C1—C11—C12	3.5 (3)	C23—C24—C25—C26	−177.5 (2)
C2—C1—C11—C12	−177.7 (2)	C29—C24—C25—C26	2.3 (3)
C16—C11—C12—C13	1.0 (3)	C24—C25—C26—C27	0.7 (4)
C1—C11—C12—C13	−176.5 (2)	C25—C26—C27—C28	−2.6 (4)
C11—C12—C13—C14	0.2 (3)	C26—C27—C28—C29	1.5 (4)
C12—C13—C14—C15	−1.1 (3)	C27—C28—C29—C24	1.5 (3)
C12—C13—C14—Br1	178.04 (17)	C27—C28—C29—C20	−179.7 (2)
C13—C14—C15—C16	0.7 (3)	C23—C24—C29—C28	176.54 (19)
Br1—C14—C15—C16	−178.38 (17)	C25—C24—C29—C28	−3.3 (3)
C14—C15—C16—C11	0.5 (3)	C23—C24—C29—C20	−2.4 (3)
C12—C11—C16—C15	−1.4 (3)	C25—C24—C29—C20	177.78 (19)
C1—C11—C16—C15	176.0 (2)	C21—C20—C29—C28	−175.28 (19)
O3—C3—C20—C21	−152.8 (2)	C3—C20—C29—C28	11.0 (3)
O2—C3—C20—C21	24.1 (3)	C21—C20—C29—C24	3.6 (3)
O3—C3—C20—C29	21.0 (3)	C3—C20—C29—C24	−170.16 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1^{i}	0.95	2.41	3.240 (3)	146
C23—H23···O3^{ii}	0.95	2.51	3.292 (3)	140

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