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3-Acetyl-7-[2-(morpholin-4-yl)ethoxy]chromen-2-one

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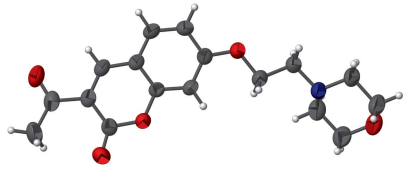
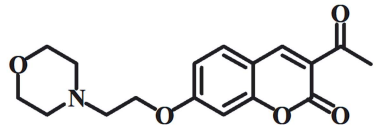
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{19}NO_5$, the morpholine ring adopts a chair conformation with the exocyclic N—C bond in an equatorial orientation. In the crystal, the molecules are linked by C—H···O and weak aromatic π–π stacking interactions, thereby generating a layered structure.

3D view



Chemical scheme



Structure description

Coumarin derivatives display many biological activities, such as antiviral, anti-HIV, anti-neoplasm and are used as fluorescent dyes (Bai & Dong, 2016). We have reported good luminescent properties and excellent cell biocompatibility (Jiao *et al.*, 2018) in coumarin derivatives. In the title compound, a morpholine ring, a typical lysosome-targeting moiety (Li *et al.*, 2018), is linked to a 7-hydroxy-3-acetylcoumarin unit *via* a flexible-chain (–O–CH_2–).

The molecular structure of the title compound is shown in Fig. 1. The coumarin ring system is essentially planar with a dihedral angle of 0.24 (5)° between the fused rings. The morpholine ring adopts a chair conformation with the exocyclic N—C bond in an equatorial orientation.

In the crystal, a one-dimensional chain-like structure is consolidated by C17—H17*A*···O4 and C17—H17*B*···O1 hydrogen bonds (Table 1) and weak aromatic π–π stacking [centroid–centroid separation = 3.6422 (10) Å] (Fig. 2). The chains are connected through very weak C18—H18*B*···O1 interactions (Fig. 3).

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

D—H···A	D—H	H···A	D···A	D—H···A
C17—H17A···O4^{i}	0.97	2.60	3.383 (2)	138
C17—H17B···O1^{ii}	0.97	2.46	3.4136 (19)	167
C18—H18B···O1^{iii}	0.97	2.68	3.459 (2)	137

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

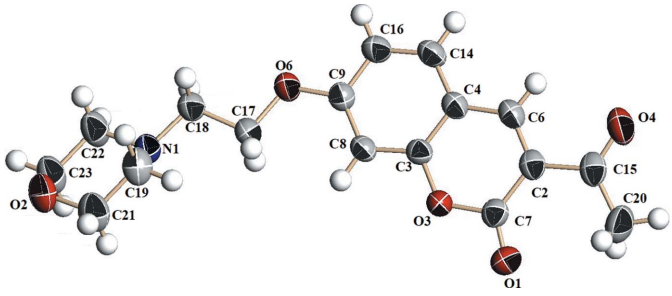


Figure 1
The molecular structure of title compound, showing displacement ellipsoids drawn at the 30% probability level.

Synthesis and crystallization

To a solution of 3-acetyl-7-hydroxy-chromen-2-one (0.50 g, 2.47 mmol) in acetonitrile (15 ml) were added potassium carbonate (1.02 g, 7.41 mmol) and 4-(2-chloro-ethyl)-morpholine (0.55 g, 2.97 mmol). The mixture was refluxed for 6 h. After completion of the reaction, the solvent was evaporated under reduced pressure. The crude compound was purified on a silica gel column (petroleum ether: ethyl acetate = 1:1 *v*/*v*) giving a yellow solid (0.65 g, 83%) and yellow block-shaped crystals were recrystallized from a petroleum ether–ethyl acetate solvent mixture.

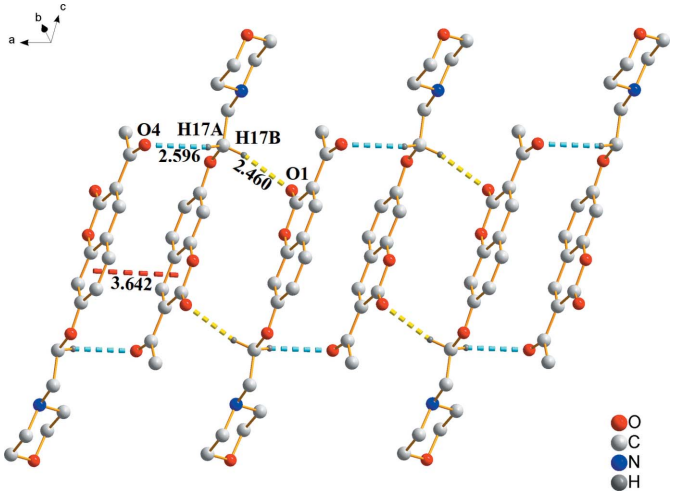


Figure 2
The crystal packing of the title compound. The weak C—H···O hydrogen bonds are shown as yellow and turquoise dashed lines, and the weak π–π stacking contacts are drawn as red dashed lines. H atoms not involved in this network have been omitted.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{19}NO_5$
M_r	317.33
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	8.4620 (14), 16.243 (3), 11.477 (2)
β (°)	93.091 (2)
V (Å^3)	1575.2 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.2 × 0.2 × 0.2
Data collection	
Diffractometer	Bruker SMART CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11443, 2916, 2427
R_{int}	0.038
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.123, 1.03
No. of reflections	2916
No. of parameters	209
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.30

Computer programs: *SMART* and *SAINT* (Bruker, 2004) and *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

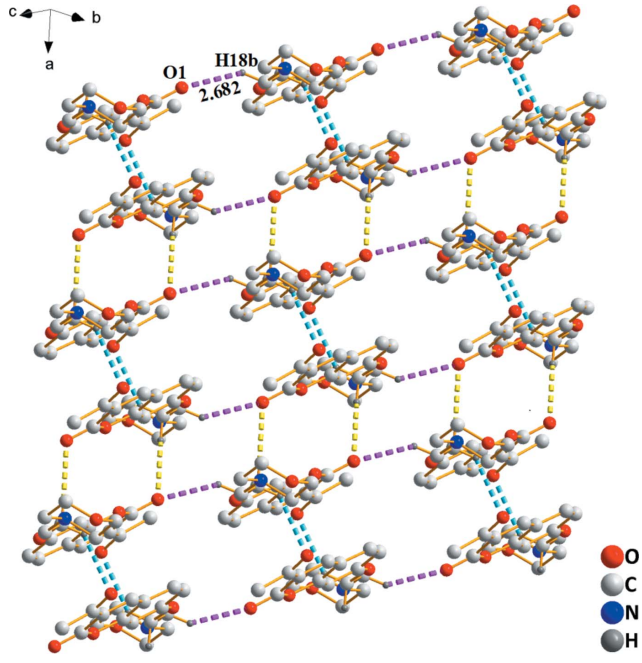


Figure 3
The sheets generated by C18—H18B···O1 (purple dashed lines) interactions. H atoms not involved in directional interactions have been omitted.

Funding information

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full crystallographic data

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Crystal data

$C_{17}H_{19}NO_5$
M_r = 317.33
Monoclinic, $P2_1/c$
a = 8.4620 (14) Å
b = 16.243 (3) Å
c = 11.477 (2) Å
β = 93.091 (2)°
V = 1575.2 (5) Å^3
Z = 4
$F(000)$ = 672
D_x = 1.338 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5468 reflections
θ = 2.5–27.1°
μ = 0.10 mm^{-1}
T = 296 K
Block, yellow
0.2 × 0.2 × 0.2 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
11443 measured reflections
2916 independent reflections
2427 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 25.5°, θ_{min} = 2.2°
h = −10→10
k = −19→19
l = −13→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.03
2916 reflections
209 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2 + 0.2289P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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_{iso}*/U_{eq}
O3	0.78260 (11)	1.00250 (6)	1.05321 (8)	0.0496 (3)
C2	0.62360 (15)	1.09671 (8)	1.16169 (12)	0.0462 (3)
C3	0.71768 (15)	1.03229 (8)	0.94956 (11)	0.0420 (3)
C4	0.60334 (15)	1.09440 (8)	0.94933 (12)	0.0446 (3)
O6	0.73725 (13)	0.99507 (6)	0.63799 (8)	0.0571 (3)
C6	0.55908 (16)	1.12462 (8)	1.05895 (12)	0.0476 (3)
H6	0.4822	1.1655	1.0601	0.057*
C7	0.74346 (16)	1.03202 (9)	1.16160 (12)	0.0479 (3)
C8	0.76948 (15)	0.99760 (8)	0.84890 (11)	0.0449 (3)
H8	0.8468	0.9568	0.8518	0.054*
C9	0.70248 (16)	1.02551 (8)	0.74304 (12)	0.0466 (3)
O1	0.81172 (13)	0.99990 (7)	1.24383 (9)	0.0643 (3)
N1	0.94110 (13)	0.82784 (7)	0.49385 (10)	0.0480 (3)
O4	0.46331 (15)	1.18477 (7)	1.26493 (11)	0.0730 (4)
O2	1.02784 (15)	0.67139 (7)	0.40415 (11)	0.0727 (4)
C14	0.54063 (18)	1.12231 (9)	0.84065 (13)	0.0535 (4)
H14	0.4653	1.1640	0.8375	0.064*
C15	0.56956 (17)	1.13410 (9)	1.27201 (13)	0.0541 (4)
C16	0.58914 (18)	1.08882 (9)	0.73952 (13)	0.0558 (4)
H16	0.5470	1.1080	0.6680	0.067*
C17	0.84068 (16)	0.92492 (9)	0.63441 (12)	0.0508 (3)
H17A	0.8035	0.8809	0.6830	0.061*
H17B	0.9473	0.9397	0.6619	0.061*
C18	0.83778 (18)	0.89834 (10)	0.50911 (12)	0.0559 (4)
H18A	0.7304	0.8840	0.4830	0.067*
H18B	0.8716	0.9437	0.4615	0.067*
C19	0.8765 (2)	0.75130 (9)	0.53613 (15)	0.0635 (4)
H19A	0.7783	0.7386	0.4923	0.076*
H19B	0.8538	0.7571	0.6177	0.076*
C20	0.6433 (2)	1.11053 (14)	1.38787 (15)	0.0812 (6)
H20A	0.5949	1.1412	1.4479	0.122*
H20B	0.7545	1.1225	1.3899	0.122*
H20C	0.6281	1.0527	1.4005	0.122*
C21	0.9933 (2)	0.68219 (10)	0.52273 (17)	0.0733 (5)
H21A	1.0902	0.6944	0.5685	0.088*
H21B	0.9498	0.6315	0.5523	0.088*
C22	0.9760 (2)	0.81673 (11)	0.37205 (15)	0.0721 (5)
H22A	1.0234	0.8665	0.3429	0.087*
H22B	0.8787	0.8063	0.3258	0.087*
C23	1.0882 (2)	0.74535 (11)	0.36046 (16)	0.0733 (5)

H23A	1.1090	0.7380	0.2788	0.088*
H23B	1.1879	0.7580	0.4023	0.088*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0526 (5)	0.0503 (6)	0.0454 (5)	0.0114 (4)	−0.0014 (4)	−0.0040 (4)
C2	0.0437 (7)	0.0437 (7)	0.0516 (7)	−0.0062 (5)	0.0063 (5)	−0.0074 (6)
C3	0.0411 (7)	0.0383 (7)	0.0464 (7)	0.0002 (5)	−0.0004 (5)	−0.0005 (5)
C4	0.0433 (7)	0.0388 (7)	0.0516 (8)	0.0021 (5)	0.0035 (6)	−0.0021 (6)
O6	0.0705 (7)	0.0564 (6)	0.0450 (5)	0.0185 (5)	0.0066 (5)	−0.0004 (4)
C6	0.0440 (7)	0.0402 (7)	0.0591 (7)	0.0026 (5)	0.0070 (6)	−0.0055 (6)
C7	0.0466 (7)	0.0495 (8)	0.0475 (8)	−0.0036 (6)	0.0022 (6)	−0.0048 (6)
C8	0.0433 (7)	0.0404 (7)	0.0511 (8)	0.0062 (5)	0.0025 (6)	−0.0026 (6)
C9	0.0503 (7)	0.0435 (7)	0.0464 (7)	0.0011 (6)	0.0063 (6)	−0.0006 (6)
O1	0.0696 (7)	0.0736 (7)	0.0489 (6)	0.0128 (6)	−0.0047 (5)	0.0008 (5)
N1	0.0522 (6)	0.0453 (6)	0.0472 (6)	0.0026 (5)	0.0087 (5)	−0.0012 (5)
O4	0.0785 (8)	0.0678 (7)	0.0741 (8)	0.0116 (6)	0.0158 (6)	−0.0191 (6)
O2	0.0882 (8)	0.0531 (7)	0.0777 (8)	0.0008 (6)	0.0135 (6)	−0.0180 (5)
C14	0.0559 (8)	0.0464 (7)	0.0584 (8)	0.0143 (6)	0.0045 (6)	0.0032 (6)
C15	0.0514 (8)	0.0526 (8)	0.0593 (9)	−0.0098 (7)	0.0114 (6)	−0.0127 (7)
C16	0.0636 (9)	0.0535 (8)	0.0503 (8)	0.0139 (7)	0.0017 (6)	0.0072 (6)
C17	0.0499 (8)	0.0516 (8)	0.0510 (8)	0.0085 (6)	0.0041 (6)	−0.0037 (6)
C18	0.0613 (9)	0.0596 (9)	0.0471 (8)	0.0132 (7)	0.0070 (6)	0.0000 (7)
C19	0.0688 (10)	0.0562 (9)	0.0672 (10)	−0.0113 (7)	0.0190 (8)	−0.0057 (7)
C20	0.0825 (12)	0.1098 (15)	0.0519 (9)	0.0095 (11)	0.0090 (8)	−0.0199 (10)
C21	0.0969 (13)	0.0463 (9)	0.0784 (12)	−0.0018 (8)	0.0191 (10)	−0.0012 (8)
C22	0.0938 (13)	0.0686 (10)	0.0565 (9)	0.0241 (9)	0.0272 (8)	0.0052 (8)
C23	0.0868 (12)	0.0658 (10)	0.0699 (10)	0.0166 (9)	0.0282 (9)	−0.0022 (8)

Geometric parameters (Å, º)

O3—C3	1.3714 (15)	C14—C16	1.365 (2)
O3—C7	1.3898 (16)	C14—H14	0.9300
C2—C6	1.351 (2)	C15—C20	1.488 (2)
C2—C7	1.4603 (19)	C16—H16	0.9300
C2—C15	1.4979 (19)	C17—C18	1.5003 (19)
C3—C8	1.3780 (18)	C17—H17A	0.9700
C3—C4	1.3978 (18)	C17—H17B	0.9700
C4—C14	1.404 (2)	C18—H18A	0.9700
C4—C6	1.4195 (19)	C18—H18B	0.9700
O6—C9	1.3502 (16)	C19—C21	1.509 (2)
O6—C17	1.4388 (16)	C19—H19A	0.9700
C6—H6	0.9300	C19—H19B	0.9700
C7—O1	1.1991 (17)	C20—H20A	0.9600
C8—C9	1.3888 (19)	C20—H20B	0.9600
C8—H8	0.9300	C20—H20C	0.9600
C9—C16	1.4053 (19)	C21—H21A	0.9700

N1—C19	1.4519 (19)	C21—H21B	0.9700
N1—C22	1.4555 (19)	C22—C23	1.509 (2)
N1—C18	1.4571 (17)	C22—H22A	0.9700
O4—C15	1.2184 (18)	C22—H22B	0.9700
O2—C23	1.408 (2)	C23—H23A	0.9700
O2—C21	1.418 (2)	C23—H23B	0.9700

C3—O3—C7	123.44 (11)	O6—C17—H17B	110.5
C6—C2—C7	119.27 (12)	C18—C17—H17B	110.5
C6—C2—C15	118.31 (13)	H17A—C17—H17B	108.7
C7—C2—C15	122.42 (13)	N1—C18—C17	111.25 (11)
O3—C3—C8	116.89 (11)	N1—C18—H18A	109.4
O3—C3—C4	120.08 (11)	C17—C18—H18A	109.4
C8—C3—C4	123.03 (12)	N1—C18—H18B	109.4
C3—C4—C14	117.59 (12)	C17—C18—H18B	109.4
C3—C4—C6	117.62 (12)	H18A—C18—H18B	108.0
C14—C4—C6	124.79 (12)	N1—C19—C21	110.06 (13)
C9—O6—C17	118.54 (11)	N1—C19—H19A	109.6
C2—C6—C4	122.96 (13)	C21—C19—H19A	109.6
C2—C6—H6	118.5	N1—C19—H19B	109.6
C4—C6—H6	118.5	C21—C19—H19B	109.6
O1—C7—O3	115.23 (12)	H19A—C19—H19B	108.2
O1—C7—C2	128.14 (13)	C15—C20—H20A	109.5
O3—C7—C2	116.62 (12)	C15—C20—H20B	109.5
C3—C8—C9	117.81 (12)	H20A—C20—H20B	109.5
C3—C8—H8	121.1	C15—C20—H20C	109.5
C9—C8—H8	121.1	H20A—C20—H20C	109.5
O6—C9—C8	124.29 (12)	H20B—C20—H20C	109.5
O6—C9—C16	115.06 (12)	O2—C21—C19	111.09 (15)
C8—C9—C16	120.65 (12)	O2—C21—H21A	109.4
C19—N1—C22	108.32 (12)	C19—C21—H21A	109.4
C19—N1—C18	113.20 (11)	O2—C21—H21B	109.4
C22—N1—C18	111.54 (11)	C19—C21—H21B	109.4
C23—O2—C21	109.50 (12)	H21A—C21—H21B	108.0
C16—C14—C4	120.67 (13)	N1—C22—C23	109.95 (14)
C16—C14—H14	119.7	N1—C22—H22A	109.7
C4—C14—H14	119.7	C23—C22—H22A	109.7
O4—C15—C20	120.38 (14)	N1—C22—H22B	109.7
O4—C15—C2	118.38 (14)	C23—C22—H22B	109.7
C20—C15—C2	121.24 (14)	H22A—C22—H22B	108.2
C14—C16—C9	120.22 (13)	O2—C23—C22	112.45 (14)
C14—C16—H16	119.9	O2—C23—H23A	109.1
C9—C16—H16	119.9	C22—C23—H23A	109.1
O6—C17—C18	106.06 (11)	O2—C23—H23B	109.1
O6—C17—H17A	110.5	C22—C23—H23B	109.1
C18—C17—H17A	110.5	H23A—C23—H23B	107.8

C7—O3—C3—C8	179.24 (11)	C3—C4—C14—C16	0.8 (2)

C7—O3—C3—C4	−1.41 (19)	C6—C4—C14—C16	−179.21 (13)
O3—C3—C4—C14	−179.79 (12)	C6—C2—C15—O4	4.1 (2)
C8—C3—C4—C14	−0.5 (2)	C7—C2—C15—O4	−176.30 (13)
O3—C3—C4—C6	0.20 (19)	C6—C2—C15—C20	−175.74 (14)
C8—C3—C4—C6	179.51 (12)	C7—C2—C15—C20	3.8 (2)
C7—C2—C6—C4	−0.6 (2)	C4—C14—C16—C9	0.2 (2)
C15—C2—C6—C4	179.04 (11)	O6—C9—C16—C14	177.72 (14)
C3—C4—C6—C2	0.8 (2)	C8—C9—C16—C14	−1.6 (2)
C14—C4—C6—C2	−179.25 (13)	C9—O6—C17—C18	171.99 (12)
C3—O3—C7—O1	−179.41 (12)	C19—N1—C18—C17	74.45 (16)
C3—O3—C7—C2	1.58 (18)	C22—N1—C18—C17	−163.10 (14)
C6—C2—C7—O1	−179.44 (14)	O6—C17—C18—N1	179.07 (11)
C15—C2—C7—O1	1.0 (2)	C22—N1—C19—C21	58.51 (18)
C6—C2—C7—O3	−0.59 (18)	C18—N1—C19—C21	−177.26 (13)
C15—C2—C7—O3	179.84 (11)	C23—O2—C21—C19	57.98 (19)
O3—C3—C8—C9	178.49 (11)	N1—C19—C21—O2	−59.95 (19)
C4—C3—C8—C9	−0.8 (2)	C19—N1—C22—C23	−57.02 (18)
C17—O6—C9—C8	5.1 (2)	C18—N1—C22—C23	177.77 (14)
C17—O6—C9—C16	−174.15 (12)	C21—O2—C23—C22	−57.3 (2)
C3—C8—C9—O6	−177.39 (12)	N1—C22—C23—O2	57.8 (2)
C3—C8—C9—C16	1.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*A*···O4^{i}	0.97	2.60	3.383 (2)	138
C17—H17*B*···O1^{ii}	0.97	2.46	3.4136 (19)	167
C18—H18*B*···O1^{iii}	0.97	2.68	3.459 (2)	137

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