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N-(2-Hydroxyethyl)-1,8-naphthalimide

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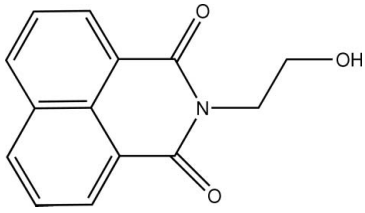
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.054; wR factor = 0.208; data-to-parameter ratio = 12.2.

In the molecule of the title compound, $C_{14}H_{11}NO_3$, the naphthalimide ring system is nearly planar (r.m.s. deviation 0.0139 Å). In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules into centrosymmetric dimers forming $R_2^2(14)$ ring motifs. π–π contacts between the naphthalimide rings [centroid–centroid distances = 3.648 (3), 3.783 (3), 3.635 (3), 3.722 (3) and 3.755 (3) Å] may further stabilize the structure.

Related literature

For a related structure, see: Prezhdo *et al.* (2007). For bond-length data, see: Allen *et al.* (1987). For ring-motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{11}NO_3$
M_r = 241.24
Triclinic, $P\overline{1}$
a = 7.5480 (15) Å
b = 8.8300 (18) Å
c = 10.101 (2) Å
α = 96.760 (19)°
β = 109.94 (3)°
γ = 114.60 (3)°
V = 548.2 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.970, T_{max} = 0.990
2159 measured reflections
1995 independent reflections
1330 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.208
S = 1.00
1995 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.82	1.97	2.771 (4)	165

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2674).

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N-(2-Hydroxyethyl)-1,8-naphthalimide

Jie Sun, Ai-lin Yuan, Hai-Bo Wang and Jie Sun

S1. Comment

As part of our ongoing studies on N-substituted 1,8-naphthalimides (Prezhdo *et al.*, 2007), we report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (N/C3-C5/C10/C11), B (C5-C10) and C (C9-C14) are, of course, planar, and they are oriented at dihedral angles of A/B = 1.79 (3), A/C = 1.14 (3) and B/C = 1.00 (3) °. So, they are nearly coplanar. Intramolecular C-H···O interaction (Table 1) results in the formation of a five-membered ring D (O2/N/C2/C3/H2A), having envelope conformation, with atom H2A displaced by -0.302 (3) Å from the plane of the other ring atoms.

In the crystal structure, intermolecular O-H···O hydrogen bonds (Table 1) link the molecules into centrosymmetric dimers forming $R_2^2(14)$ ring motifs (Fig. 2) (Bernstein *et al.*, 1996), in which they may be effective in the stabilization of the structure. The π–π contacts between the naphthalimide rings, Cg1—Cg1^{i}, Cg1—Cg2^{i}, Cg1—Cg3^{ii}, Cg2—Cg3^{ii} and Cg3—Cg3^{ii} [symmetry codes: (i) 1 - x, 1 - y, 1 - z, (ii) 2 - x, 1 - y, 1 - z, where Cg1, Cg2 and Cg3 are centroids of the rings A (N/C3-C5/C10/C11), B (C5-C10) and C (C9-C14), respectively] may further stabilize the structure, with centroid-centroid distances of 3.648 (3), 3.783 (3), 3.635 (3), 3.722 (3) and 3.755 (3) Å, respectively.

S2. Experimental

For the preparation of the title compound, 1,8-naphthalic anhydride (1.98 g, 0.01 mol) and 2-aminoethanol (0.02 mol) were mixed with acetic acid (50 ml). The reaction mixture was refluxed for 8 h, and then poured into cold water. The resulting solids were filtered off. The solid products were boiled with an aqueous solution of sodium bicarbonate (10%, 50 ml) for 20 min, and the insoluble solid residues were dried in vacuo. Column chromatography on aluminium oxide with the C_6H_6 eluent gave light-brown solution. Crystals suitable for X-ray analysis were obtained by slow evaporation of an acetone solution (yield; 96%, m.p. 413 K).

S3. Refinement

H atoms were positioned geometrically, with O-H = 0.82 Å (for OH) and C-H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,O), where x = 1.5 for OH H and x = 1.2 for all other H atoms.

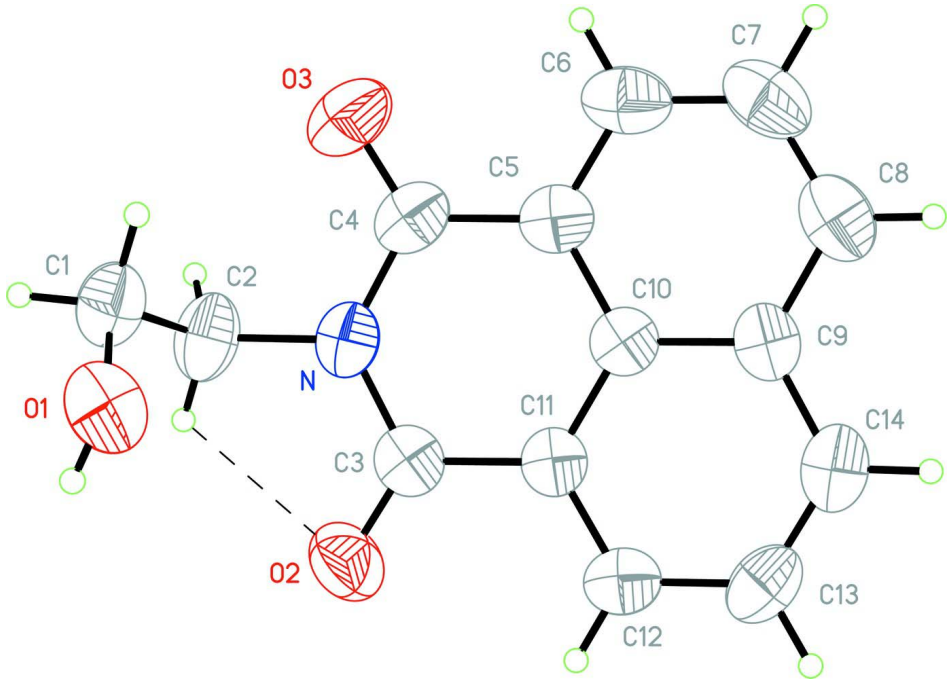

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bond is shown as dashed line.

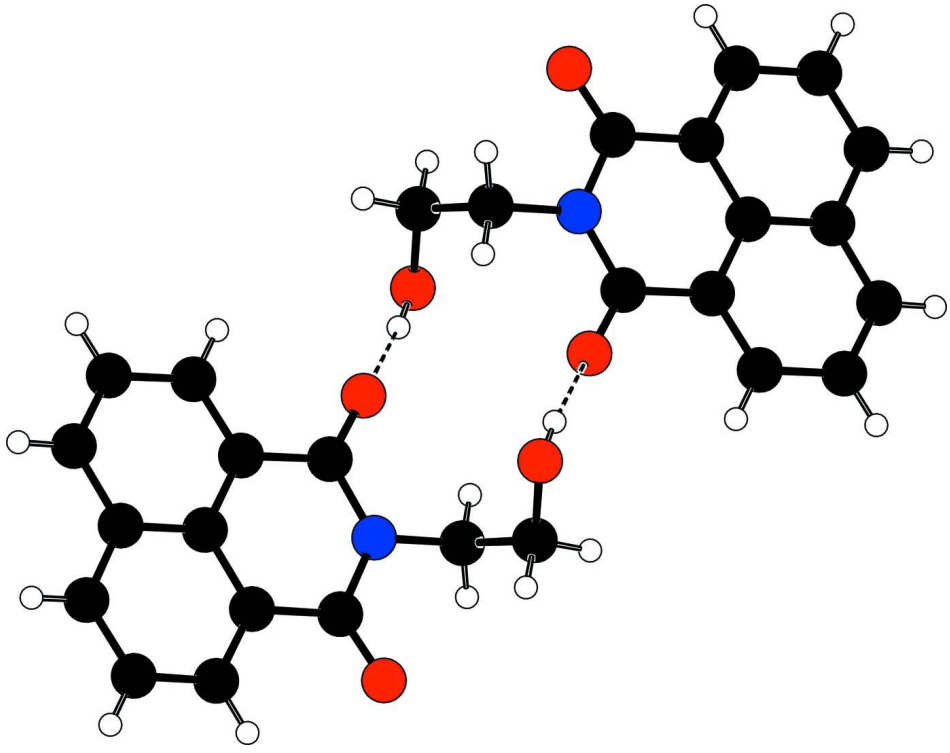

Figure 2

A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

N-(2-Hydroxyethyl)naphthalene-1,8-dicarboximide

Crystal data

$C_{14}H_{11}NO_3$
$M_r = 241.24$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.5480$ (15) Å
$b = 8.8300$ (18) Å
$c = 10.101$ (2) Å
$\alpha = 96.760$ (19)°
$\beta = 109.94$ (3)°
$\gamma = 114.60$ (3)°
$V = 548.2$ (3) Å^3
$Z = 2$
$F(000) = 252$
$D_x = 1.461$ Mg m^{-3}
Melting point: 413 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–13°
$\mu = 0.10$ mm^{-1}
$T = 294$ K
Block, green
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.970$, $T_{max} = 0.990$
2159 measured reflections
1995 independent reflections
1330 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.3°$
$h = 0 \rightarrow 9$
$k = -10 \rightarrow 9$
$l = -12 \rightarrow 11$
3 standard reflections every 120 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.208$
$S = 1.00$
1995 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.4P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.035 (10)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.4860 (4)	0.2063 (3)	−0.0242 (2)	0.0624 (7)

H1A	0.4763	0.2711	−0.0749	0.094*
O2	0.6221 (4)	0.5936 (3)	0.1937 (3)	0.0677 (8)
O3	0.2875 (4)	0.1029 (3)	0.3070 (3)	0.0729 (8)
N	0.4553 (4)	0.3481 (3)	0.2496 (3)	0.0480 (7)
C1	0.3020 (6)	0.1341 (5)	0.0027 (4)	0.0599 (9)
H1B	0.3052	0.0455	0.0504	0.072*
H1C	0.1746	0.0767	−0.0913	0.072*
C2	0.2818 (5)	0.2660 (5)	0.0982 (4)	0.0610 (10)
H2A	0.2828	0.3566	0.0522	0.073*
H2B	0.1439	0.2081	0.1026	0.073*
C3	0.6193 (5)	0.5166 (4)	0.2858 (3)	0.0472 (8)
C4	0.4392 (5)	0.2495 (4)	0.3487 (4)	0.0509 (8)
C5	0.6124 (5)	0.3314 (4)	0.5006 (3)	0.0464 (8)
C6	0.6103 (6)	0.2394 (5)	0.6016 (4)	0.0601 (10)
H6A	0.4975	0.1263	0.5743	0.072*
C7	0.7759 (7)	0.3145 (5)	0.7442 (4)	0.0677 (11)
H7A	0.7726	0.2510	0.8114	0.081*
C8	0.9433 (6)	0.4804 (5)	0.7870 (4)	0.0601 (9)
H8A	1.0531	0.5286	0.8826	0.072*
C9	0.9504 (5)	0.5784 (4)	0.6872 (3)	0.0450 (7)
C10	0.7830 (5)	0.5027 (4)	0.5413 (3)	0.0407 (7)
C11	0.7899 (5)	0.5973 (4)	0.4390 (3)	0.0423 (7)
C12	0.9566 (5)	0.7653 (4)	0.4817 (4)	0.0494 (8)
H12A	0.9611	0.8282	0.4142	0.059*
C13	1.1206 (5)	0.8421 (4)	0.6277 (4)	0.0560 (9)
H13A	1.2317	0.9562	0.6562	0.067*
C14	1.1181 (5)	0.7516 (5)	0.7266 (4)	0.0536 (9)
H14A	1.2283	0.8041	0.8222	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0681 (16)	0.0792 (17)	0.0581 (15)	0.0486 (14)	0.0293 (12)	0.0251 (12)
O2	0.0783 (17)	0.0742 (17)	0.0564 (15)	0.0431 (14)	0.0243 (13)	0.0308 (13)
O3	0.0595 (16)	0.0526 (15)	0.0854 (19)	0.0114 (13)	0.0330 (14)	0.0078 (13)
N	0.0442 (15)	0.0521 (15)	0.0468 (15)	0.0266 (13)	0.0177 (12)	0.0061 (12)
C1	0.057 (2)	0.059 (2)	0.053 (2)	0.0252 (17)	0.0200 (16)	0.0039 (16)
C2	0.0468 (19)	0.068 (2)	0.055 (2)	0.0298 (17)	0.0112 (16)	0.0000 (17)
C3	0.0512 (19)	0.0525 (18)	0.0517 (19)	0.0341 (16)	0.0260 (15)	0.0161 (15)
C4	0.0480 (19)	0.0451 (18)	0.063 (2)	0.0226 (16)	0.0310 (16)	0.0069 (15)
C5	0.0514 (18)	0.0469 (17)	0.0566 (19)	0.0302 (15)	0.0327 (16)	0.0148 (15)
C6	0.078 (2)	0.056 (2)	0.075 (3)	0.0383 (19)	0.054 (2)	0.0285 (18)
C7	0.095 (3)	0.081 (3)	0.063 (2)	0.057 (2)	0.049 (2)	0.039 (2)
C8	0.073 (2)	0.081 (3)	0.051 (2)	0.052 (2)	0.0338 (18)	0.0238 (18)
C9	0.0469 (17)	0.0588 (19)	0.0434 (17)	0.0359 (16)	0.0232 (14)	0.0123 (14)
C10	0.0421 (16)	0.0460 (16)	0.0483 (17)	0.0291 (14)	0.0260 (14)	0.0120 (13)
C11	0.0455 (17)	0.0447 (16)	0.0463 (17)	0.0296 (14)	0.0214 (14)	0.0119 (13)
C12	0.0551 (19)	0.0443 (17)	0.062 (2)	0.0293 (15)	0.0328 (16)	0.0171 (15)

C13	0.0434 (18)	0.0478 (18)	0.068 (2)	0.0186 (15)	0.0238 (17)	0.0021 (17)
C14	0.0481 (18)	0.065 (2)	0.0490 (19)	0.0339 (17)	0.0190 (15)	0.0025 (16)

Geometric parameters (Å, º)

O1—C1	1.403 (4)	C6—C7	1.391 (5)
O1—H1A	0.8200	C6—H6A	0.9300
O2—C3	1.216 (4)	C7—C8	1.366 (5)
O3—C4	1.214 (4)	C7—H7A	0.9300
N—C2	1.470 (4)	C8—C9	1.404 (5)
N—C3	1.383 (4)	C8—H8A	0.9300
N—C4	1.404 (4)	C9—C10	1.418 (4)
C1—C2	1.515 (5)	C9—C14	1.414 (5)
C1—H1B	0.9700	C10—C11	1.405 (4)
C1—H1C	0.9700	C11—C12	1.376 (4)
C2—H2A	0.9700	C12—C13	1.410 (5)
C2—H2B	0.9700	C12—H12A	0.9300
C3—C11	1.476 (4)	C13—C14	1.351 (5)
C4—C5	1.474 (5)	C13—H13A	0.9300
C5—C6	1.378 (5)	C14—H14A	0.9300
C5—C10	1.409 (4)		
C1—O1—H1A	109.5	C5—C6—H6A	119.8
C3—N—C4	124.6 (3)	C7—C6—H6A	119.8
C3—N—C2	118.3 (3)	C8—C7—C6	120.9 (3)
C4—N—C2	117.1 (3)	C8—C7—H7A	119.6
O1—C1—C2	114.2 (3)	C6—C7—H7A	119.6
O1—C1—H1B	108.7	C7—C8—C9	120.4 (3)
C2—C1—H1B	108.7	C7—C8—H8A	119.8
O1—C1—H1C	108.7	C9—C8—H8A	119.8
C2—C1—H1C	108.7	C8—C9—C14	122.6 (3)
H1B—C1—H1C	107.6	C8—C9—C10	119.2 (3)
N—C2—C1	113.5 (3)	C14—C9—C10	118.2 (3)
N—C2—H2A	108.9	C11—C10—C5	120.9 (3)
C1—C2—H2A	108.9	C11—C10—C9	120.0 (3)
N—C2—H2B	108.9	C5—C10—C9	119.1 (3)
C1—C2—H2B	108.9	C12—C11—C10	119.9 (3)
H2A—C2—H2B	107.7	C12—C11—C3	120.0 (3)
O2—C3—N	120.7 (3)	C10—C11—C3	120.1 (3)
O2—C3—C11	121.9 (3)	C11—C12—C13	120.1 (3)
N—C3—C11	117.4 (3)	C11—C12—H12A	119.9
O3—C4—N	119.8 (3)	C13—C12—H12A	119.9
O3—C4—C5	123.0 (3)	C14—C13—C12	120.7 (3)
N—C4—C5	117.2 (3)	C14—C13—H13A	119.7
C6—C5—C10	120.1 (3)	C12—C13—H13A	119.7
C6—C5—C4	120.1 (3)	C13—C14—C9	121.1 (3)
C10—C5—C4	119.8 (3)	C13—C14—H14A	119.4
C5—C6—C7	120.4 (3)	C9—C14—H14A	119.4

C3—N—C2—C1	103.2 (4)	C4—C5—C10—C11	0.4 (4)
C4—N—C2—C1	−78.1 (4)	C6—C5—C10—C9	−0.4 (4)
O1—C1—C2—N	−64.6 (4)	C4—C5—C10—C9	−178.9 (2)
C4—N—C3—O2	180.0 (3)	C8—C9—C10—C11	−178.6 (3)
C2—N—C3—O2	−1.4 (4)	C14—C9—C10—C11	1.9 (4)
C4—N—C3—C11	0.5 (4)	C8—C9—C10—C5	0.7 (4)
C2—N—C3—C11	179.1 (2)	C14—C9—C10—C5	−178.9 (2)
C3—N—C4—O3	179.1 (3)	C5—C10—C11—C12	179.1 (2)
C2—N—C4—O3	0.5 (4)	C9—C10—C11—C12	−1.6 (4)
C3—N—C4—C5	−1.2 (4)	C5—C10—C11—C3	−1.1 (4)
C2—N—C4—C5	−179.9 (2)	C9—C10—C11—C3	178.1 (2)
O3—C4—C5—C6	2.0 (5)	O2—C3—C11—C12	1.0 (4)
N—C4—C5—C6	−177.7 (3)	N—C3—C11—C12	−179.6 (3)
O3—C4—C5—C10	−179.6 (3)	O2—C3—C11—C10	−178.8 (3)
N—C4—C5—C10	0.8 (4)	N—C3—C11—C10	0.7 (4)
C10—C5—C6—C7	0.1 (5)	C10—C11—C12—C13	0.2 (4)
C4—C5—C6—C7	178.5 (3)	C3—C11—C12—C13	−179.5 (3)
C5—C6—C7—C8	0.0 (5)	C11—C12—C13—C14	0.9 (4)
C6—C7—C8—C9	0.2 (5)	C12—C13—C14—C9	−0.6 (5)
C7—C8—C9—C14	178.9 (3)	C8—C9—C14—C13	179.7 (3)
C7—C8—C9—C10	−0.6 (5)	C10—C9—C14—C13	−0.8 (4)
C6—C5—C10—C11	178.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{i}	0.82	1.97	2.771 (4)	165
C2—H2*A*···O2	0.97	2.31	2.714 (5)	104

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.