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Methyl 2-(4-chloro-3,5-dinitrobenzamido)acetate

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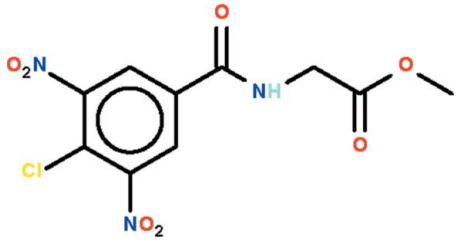
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.074; data-to-parameter ratio = 11.6.

The title molecule, $C_{10}H_8ClN_3O_7$, is twisted with the dihedral angle between the amide and benzene ring being 38.75 (11)°. The C—N—C—C torsion angle between the amide and acetyl groups is −150.1 (2)°. Finally, each nitro group is twisted out of the plane of the benzene ring to which it is connected [O—N—C—C torsion angles = 34.0 (3) and −64.5 (3)°]. Linear supramolecular chains along [010] and mediated by N—H···O hydrogen bonds between successive amide groups dominate the crystal packing. The chains are consolidated into the three-dimensional structure by C—H···O contacts.

Related literature

For biological and crystal engineering studies of related compounds, see: Liu *et al.* (2009); Eissmann & Weber (2011).



Experimental

Crystal data

$C_{10}H_8ClN_3O_7$
M_r = 317.64
Orthorhombic, $Pna2_1$
a = 14.5219 (5) Å
b = 4.7949 (2) Å
c = 18.5368 (6) Å
V = 1290.74 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 100 K
0.30 × 0.20 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.906, T_{max} = 0.967
4743 measured reflections
2258 independent reflections
2134 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.08
2258 reflections
194 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 725 Friedel pairs
Flack parameter: −0.05 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.88 (1)	1.99 (1)	2.833 (3)	163 (3)
C1—H1a···O7^{ii}	0.98	2.59	3.460 (3)	148
C3—H3a···O6^{iii}	0.99	2.53	3.502 (3)	169
C3—H3b···O2^{iv}	0.99	2.42	3.380 (3)	162
C10—H10···O5^{v}	0.95	2.37	3.223 (3)	149

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5145).

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supporting information

Acta Cryst. (2011). E**67**, o3486 [https://doi.org/10.1107/S1600536811050446]

Methyl 2-(4-chloro-3,5-dinitrobenzamido)acetate

Xiang-Xiang Wu, Xue-Fen Wu, Yi-Min Hou, Seik Weng Ng and Edward R. T. Tiekink

S1. Comment

Molecules related to the title compound, (I), attract interest for their biological properties (Liu *et al.*, 2009) and also in terms of crystal engineering endeavours (Eissmann & Weber, 2011). In (I), Fig. 1, the dihedral angle between the amide (O3,N1,C4) atoms and the benzene ring is 38.75 (11)°. The acetyl group is also twisted out of the plane of the amide group with the C4—N1—C3—C2 torsion angle being -150.1 (2)°. Each nitro group is twisted out of the plane of the benzene ring to which it is connected with the O4—N2—C7—C6 torsion angle = 34.0 (3)° and with O6—N3—C9—C8 = -64.5 (3)°.

The crystal packing is dominated by the formation of linear supramolecular chains along the *b* axis and mediated by N—H···O hydrogen bonds involving the amide group, Fig. 2 and Table 1. Chains are consolidated in the crystal packing by C—H···O interactions, Fig. 3 and Table 1.

S2. Experimental

To a solution of 4-chloro-3,5-dinitrobenzoic acid (0.48 g, 2 mmol) in dichloromethane (30 ml) was added 1-ethyl-3-(3-dimethylaminopropyl)carbodiimidehydrochloride (0.40 g, 2.1 mmol) and *N*,*N*-dimethylaminopyridine (25 mg, 0.2 mmol). The mixture was stirred at room temperature for an hour. Methyl 2-aminoacetate (178 mg, 2 mmol) in chloroform (20 ml) along with several drops of triethylamine were added. After another six hours, the mixture was subjected to chromatography (petroleum ether/acetone 4:1) to provide the product as a yellow solid (501.5 mg, 80% yield). Crystals were grown from a mixture of dichloromethane and *n*-hexane (1:1 *v*/*v*).

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.99 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation. The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint of N—H 0.88±0.01 Å, and with free U_{iso}.

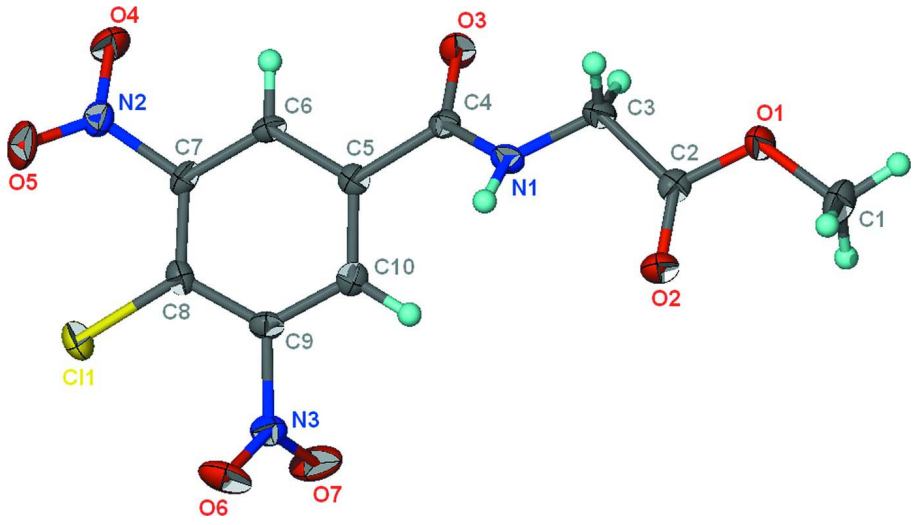

Figure 1

Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at the 70% probability level.

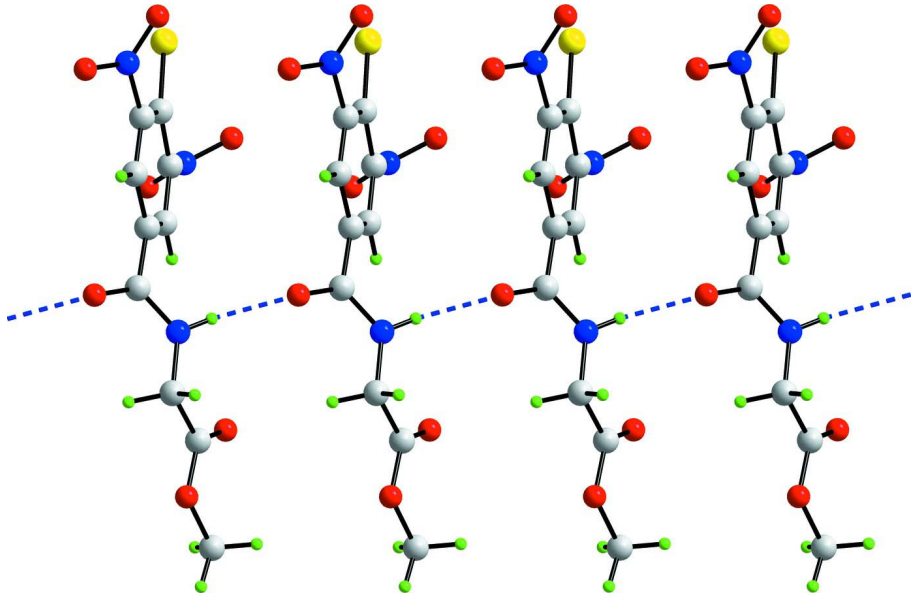

Figure 2

Supramolecular linear chain along the *b* axis in (I). The N—H···O contacts are shown as blue dashed lines.

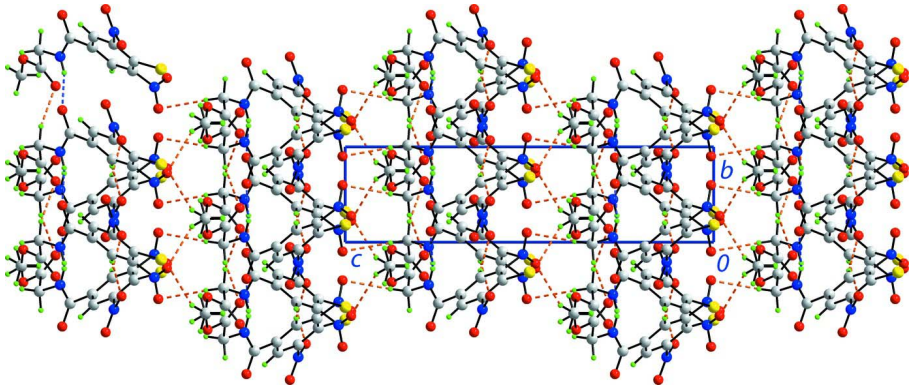

Figure 3

A view of the unit-cell contents of (I) in projection down the *a* axis. The N—H···O and C—H···O interactions are shown as blue and orange dashed lines, respectively.

Methyl 2-(4-chloro-3,5-dinitrobenzamido)acetate

Crystal data

$C_{10}H_8ClN_3O_7$
$M_r = 317.64$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 14.5219$ (5) Å
$b = 4.7949$ (2) Å
$c = 18.5368$ (6) Å
$V = 1290.74$ (8) Å^3
$Z = 4$
$F(000) = 648$
$D_x = 1.635$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2633 reflections
$\theta = 2.6$–$27.5°$
$\mu = 0.34$ mm^{-1}
$T = 100$ K
Prism, yellow
0.30 × 0.20 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
$T_{min} = 0.906$, $T_{max} = 0.967$
4743 measured reflections
2258 independent reflections
2134 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.8°$
$h = -13 \rightarrow 18$
$k = -6 \rightarrow 5$
$l = -17 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.074$
$S = 1.08$
2258 reflections
194 parameters
2 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.1422P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Absolute structure: Flack (1983), 725 Friedel pairs
Absolute structure parameter: −0.05 (6)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.97666 (4)	0.68297 (13)	0.49982 (3)	0.02449 (14)
O1	0.46196 (11)	0.5789 (4)	0.87399 (9)	0.0214 (4)
O2	0.51448 (12)	0.8605 (3)	0.78629 (9)	0.0232 (4)
O3	0.73214 (11)	0.1135 (3)	0.76810 (9)	0.0212 (4)
O4	1.03518 (12)	0.0525 (4)	0.65033 (11)	0.0278 (4)
O5	1.09547 (11)	0.4311 (4)	0.60728 (11)	0.0282 (4)
O6	0.81733 (12)	1.0984 (3)	0.50745 (10)	0.0286 (4)
O7	0.71827 (12)	0.7754 (4)	0.48417 (10)	0.0329 (5)
N1	0.67321 (13)	0.5506 (4)	0.77382 (11)	0.0154 (4)
N2	1.03014 (12)	0.2929 (4)	0.62781 (11)	0.0188 (4)
N3	0.78247 (13)	0.8709 (4)	0.51797 (10)	0.0179 (4)
C1	0.37848 (17)	0.7447 (6)	0.87927 (14)	0.0260 (5)
H1A	0.3379	0.6641	0.9160	0.039*
H1B	0.3469	0.7454	0.8326	0.039*
H1C	0.3944	0.9363	0.8928	0.039*
C2	0.52388 (15)	0.6648 (5)	0.82581 (12)	0.0152 (5)
C3	0.60829 (15)	0.4829 (5)	0.83024 (12)	0.0180 (5)
H3A	0.6381	0.5085	0.8778	0.022*
H3B	0.5899	0.2847	0.8259	0.022*
C4	0.72913 (14)	0.3572 (5)	0.74666 (12)	0.0144 (4)
C5	0.79056 (16)	0.4510 (5)	0.68634 (11)	0.0141 (5)
C6	0.87941 (15)	0.3419 (5)	0.68295 (12)	0.0144 (5)
H6	0.9001	0.2141	0.7186	0.017*
C7	0.93716 (15)	0.4205 (5)	0.62750 (12)	0.0148 (4)
C8	0.90958 (15)	0.6012 (5)	0.57315 (12)	0.0154 (5)
C9	0.81934 (15)	0.6983 (5)	0.57707 (12)	0.0148 (4)
C10	0.76008 (15)	0.6285 (4)	0.63220 (12)	0.0150 (4)
H10	0.6992	0.7008	0.6332	0.018*
H1	0.688 (2)	0.722 (3)	0.7627 (15)	0.034 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0208 (2)	0.0329 (3)	0.0197 (3)	−0.0005 (2)	0.0067 (3)	0.0055 (3)
O1	0.0187 (8)	0.0221 (9)	0.0235 (9)	0.0055 (7)	0.0068 (7)	0.0057 (8)
O2	0.0229 (9)	0.0218 (9)	0.0250 (9)	0.0052 (7)	0.0007 (7)	0.0080 (8)
O3	0.0222 (8)	0.0118 (8)	0.0296 (9)	0.0021 (6)	0.0050 (8)	0.0042 (7)
O4	0.0222 (9)	0.0231 (10)	0.0381 (11)	0.0077 (7)	0.0013 (8)	0.0099 (9)
O5	0.0127 (8)	0.0264 (9)	0.0455 (11)	−0.0055 (7)	0.0033 (8)	0.0016 (9)
O6	0.0452 (10)	0.0165 (8)	0.0239 (9)	−0.0042 (8)	−0.0027 (9)	0.0078 (8)
O7	0.0313 (10)	0.0340 (11)	0.0334 (11)	−0.0022 (8)	−0.0180 (9)	0.0075 (9)
N1	0.0197 (9)	0.0095 (9)	0.0171 (9)	0.0005 (7)	0.0019 (8)	0.0020 (8)
N2	0.0140 (10)	0.0219 (11)	0.0206 (10)	−0.0005 (8)	−0.0006 (8)	0.0001 (9)
N3	0.0220 (9)	0.0187 (10)	0.0129 (9)	0.0047 (8)	0.0004 (8)	−0.0007 (8)
C1	0.0173 (11)	0.0303 (13)	0.0305 (13)	0.0060 (11)	0.0032 (11)	−0.0036 (13)

C2	0.0167 (10)	0.0155 (11)	0.0133 (11)	0.0003 (9)	−0.0011 (9)	−0.0038 (9)
C3	0.0195 (11)	0.0176 (12)	0.0171 (11)	0.0029 (9)	0.0023 (9)	0.0044 (9)
C4	0.0133 (9)	0.0149 (12)	0.0150 (10)	−0.0016 (8)	−0.0039 (9)	0.0012 (9)
C5	0.0152 (10)	0.0128 (11)	0.0142 (10)	−0.0023 (9)	−0.0013 (8)	−0.0019 (9)
C6	0.0158 (11)	0.0112 (11)	0.0161 (10)	0.0023 (9)	−0.0031 (9)	0.0005 (9)
C7	0.0111 (10)	0.0131 (10)	0.0201 (11)	0.0018 (9)	−0.0011 (9)	−0.0035 (9)
C8	0.0148 (10)	0.0163 (12)	0.0149 (10)	−0.0029 (9)	0.0026 (9)	−0.0019 (9)
C9	0.0186 (11)	0.0106 (11)	0.0151 (10)	0.0000 (9)	−0.0016 (9)	0.0011 (9)
C10	0.0154 (10)	0.0113 (10)	0.0185 (11)	0.0002 (9)	0.0001 (9)	−0.0029 (9)

Geometric parameters (Å, º)

Cl1—C8	1.718 (2)	C1—H1B	0.9800
O1—C2	1.333 (3)	C1—H1C	0.9800
O1—C1	1.453 (3)	C2—C3	1.507 (3)
O2—C2	1.198 (3)	C3—H3A	0.9900
O3—C4	1.235 (3)	C3—H3B	0.9900
O4—N2	1.228 (3)	C4—C5	1.499 (3)
O5—N2	1.218 (2)	C5—C10	1.388 (3)
O6—N3	1.218 (2)	C5—C6	1.394 (3)
O7—N3	1.213 (2)	C6—C7	1.379 (3)
N1—C4	1.331 (3)	C6—H6	0.9500
N1—C3	1.445 (3)	C7—C8	1.388 (3)
N1—H1	0.875 (10)	C8—C9	1.393 (3)
N2—C7	1.482 (3)	C9—C10	1.377 (3)
N3—C9	1.474 (3)	C10—H10	0.9500
C1—H1A	0.9800		
C2—O1—C1	116.05 (18)	C2—C3—H3B	109.4
C4—N1—C3	121.01 (19)	H3A—C3—H3B	108.0
C4—N1—H1	115 (2)	O3—C4—N1	124.0 (2)
C3—N1—H1	123 (2)	O3—C4—C5	120.2 (2)
O5—N2—O4	124.78 (19)	N1—C4—C5	115.9 (2)
O5—N2—C7	118.92 (19)	C10—C5—C6	119.5 (2)
O4—N2—C7	116.30 (18)	C10—C5—C4	122.2 (2)
O7—N3—O6	125.1 (2)	C6—C5—C4	118.15 (19)
O7—N3—C9	116.80 (19)	C7—C6—C5	119.6 (2)
O6—N3—C9	118.10 (19)	C7—C6—H6	120.2
O1—C1—H1A	109.5	C5—C6—H6	120.2
O1—C1—H1B	109.5	C6—C7—C8	122.43 (19)
H1A—C1—H1B	109.5	C6—C7—N2	115.99 (19)
O1—C1—H1C	109.5	C8—C7—N2	121.56 (19)
H1A—C1—H1C	109.5	C7—C8—C9	116.3 (2)
H1B—C1—H1C	109.5	C7—C8—Cl1	123.59 (17)
O2—C2—O1	125.1 (2)	C9—C8—Cl1	119.92 (18)
O2—C2—C3	125.4 (2)	C10—C9—C8	123.1 (2)
O1—C2—C3	109.49 (19)	C10—C9—N3	117.46 (19)
N1—C3—C2	111.18 (18)	C8—C9—N3	119.4 (2)

N1—C3—H3A	109.4	C9—C10—C5	119.1 (2)
C2—C3—H3A	109.4	C9—C10—H10	120.5
N1—C3—H3B	109.4	C5—C10—H10	120.5
C1—O1—C2—O2	−1.9 (3)	O4—N2—C7—C8	−144.0 (2)
C1—O1—C2—C3	176.48 (19)	C6—C7—C8—C9	−0.6 (3)
C4—N1—C3—C2	−150.1 (2)	N2—C7—C8—C9	177.3 (2)
O2—C2—C3—N1	−7.6 (3)	C6—C7—C8—Cl1	−174.96 (18)
O1—C2—C3—N1	174.00 (19)	N2—C7—C8—Cl1	3.0 (3)
C3—N1—C4—O3	−2.2 (3)	C7—C8—C9—C10	1.7 (3)
C3—N1—C4—C5	177.61 (19)	Cl1—C8—C9—C10	176.32 (18)
O3—C4—C5—C10	139.5 (2)	C7—C8—C9—N3	−174.6 (2)
N1—C4—C5—C10	−40.3 (3)	Cl1—C8—C9—N3	−0.1 (3)
O3—C4—C5—C6	−36.8 (3)	O7—N3—C9—C10	−59.9 (3)
N1—C4—C5—C6	143.4 (2)	O6—N3—C9—C10	118.9 (2)
C10—C5—C6—C7	2.6 (3)	O7—N3—C9—C8	116.6 (2)
C4—C5—C6—C7	179.0 (2)	O6—N3—C9—C8	−64.5 (3)
C5—C6—C7—C8	−1.6 (3)	C8—C9—C10—C5	−0.7 (3)
C5—C6—C7—N2	−179.6 (2)	N3—C9—C10—C5	175.76 (19)
O5—N2—C7—C6	−144.8 (2)	C6—C5—C10—C9	−1.5 (3)
O4—N2—C7—C6	34.0 (3)	C4—C5—C10—C9	−177.75 (19)
O5—N2—C7—C8	37.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.88 (1)	1.99 (1)	2.833 (3)	163 (3)
C1—H1a···O7^{ii}	0.98	2.59	3.460 (3)	148
C3—H3a···O6^{iii}	0.99	2.53	3.502 (3)	169
C3—H3b···O2^{iv}	0.99	2.42	3.380 (3)	162
C10—H10···O5^{v}	0.95	2.37	3.223 (3)	149

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