# (*E*)-2-Methyl-6-[(1-phenyl-1*H*-pyrazol-4-yl)methylidene]cyclohexanone

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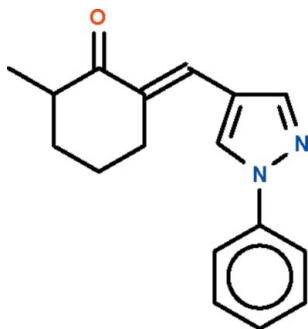Correspondence e-mail: seikweng@um.edu.my

The asymmetric unit of the title compound, $C_{17}H_{18}N_2O$, contains two independent molecules. In both, the cyclohexane ring adopts a flattened chair conformation, and the 3- and 4-methylene C atoms as well as the methyl C atoms are disordered over two positions, the occupancy of the major component being 68 (1)% in one molecule and 64 (1)% in the other. The phenyl and pyrazole rings in both molecules are approximately coplanar, the r.m.s. deviations being 0.048 and 0.015 Å, respectively. Weak intermolecular C—H⋯O hydrogen bonding is present in the crystal structure.

## Related literature

For a recent report on similar heterocylic compounds derived from substituted 1-phenylpyrazole-4-carboxaldehydes>, see: Asiri & Khan (2010).



## Experimental

### Crystal data

| | |
|---|---|
| $C_{17}H_{18}N_2O$ | $\gamma$ = 78.510 (2)° |
| $M_r$ = 266.33 | $V$ = 1379.2 (3) Å³ |
| Triclinic, $P\bar{1}$ | $Z$ = 4 |
| $a$ = 6.1152 (8) Å | Mo $K\alpha$ radiation |
| $b$ = 10.3757 (13) Å | $\mu$ = 0.08 mm⁻¹ |
| $c$ = 22.734 (3) Å | $T$ = 100 K |
| $\alpha$ = 77.542 (2)° | 0.20 × 0.20 × 0.05 mm |
| $\beta$ = 89.667 (2)° | |

### Data collection

| | |
|---|---|
| Bruker SMART APEX diffractometer | 4890 independent reflections |
| | 3235 reflections with $I > 2\sigma(I)$ |
| 14492 measured reflections | $R_{int}$ = 0.051 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.062 | 47 restraints |
| $wR(F^2)$ = 0.172 | H-atom parameters constrained |
| $S$ = 1.03 | $\Delta\rho_{max}$ = 0.40 e Å⁻³ |
| 4890 reflections | $\Delta\rho_{min}$ = −0.27 e Å⁻³ |
| 408 parameters | |

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C9—H9⋯O1[i] | 0.95 | 2.29 | 3.157 (4) | 152 |
| C26—H26⋯O2[ii] | 0.95 | 2.30 | 3.224 (4) | 164 |
| C30—H30⋯O2[ii] | 0.95 | 2.57 | 3.506 (4) | 167 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

## References

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Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

# supporting information

# (*E*)-2-Methyl-6-[(1-phenyl-1*H*-pyrazol-4-yl)methylidene]cyclohexanone

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### S1. Comment

The hydrogen atoms of the *α*-methylene carbons of cyclohexanone are relatively acidic, and the compound can undergo an aldol-type condensation with an aldehyde. This study employs the aldehyde, 1-phenylpyrazole-4-carboxaldehyde, to react with 2-methylcyclohexanone to yield a new heterocyclic compound that is expected to possess useful biological actvity. The present structural study extends a recent report on similar heterocylic compounds derived from substituted 1-phenylpyrazole-4-carboxaldehydes (Asiri & Khan, 2010). In the two independent molecules of $C_{17}H_{18}N_2O$ (Scheme I), the cyclohexane ring adopts a flattened chair conformation; the phenylpyrazolyl substituent of the ring in both molecules, which is planar (r.m.s. deviation 0.048, 0.015 Å), is approximately co-planar with the mean plane passing through the cyclohexane ring (Fig.1). The methylene carbon that provided the acidic H atoms is $sp^2$-hybridized in the product; the strain in the ring is now reflected in an ethylene fragment as well as in the methyl substituent adopting two orientations. The 3- and 4-methylene as well as the methyl C atoms are disordered, with the occupancy of the major component being 68 (1)% in one molecule and 64 (1)% in the other.

### S2. Experimental

1-Phenylpyrazole-4-carboxaldehyde (1.72 g, 0.01 mol) in ethanol (20 ml) was added to 2-methylcyclohexanone (1.12 g, 0.01 mol) in 20% alcoholic potassium hydroxide (20 ml). The mixture was stirred for 6 h. The solid product that separated was collected and recrystallized from ethanol.

### S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95 to 1.00 Å) and were included in the refinement in the riding model approximation, with $U$(H) set to 1.2 to $1.5U_{eq}$(C).

The 3- and 4-methylene as well as the methyl substituent of the cyclohexane ring is disordered over two positions; for one molecule, the occupancy of the major component refined to 68 (1)% and for the other, this refined to 64 (1)%. The carbon-carbon single-bond distances were restrained to 1.54–0.01 Å; for the methyl C atom, the 1,3-related distances were restrained to 2.51±0.01 Å. Additionally, the anisotropic temperature factors of the minor-component atoms were restrained to be nearly isotropic.
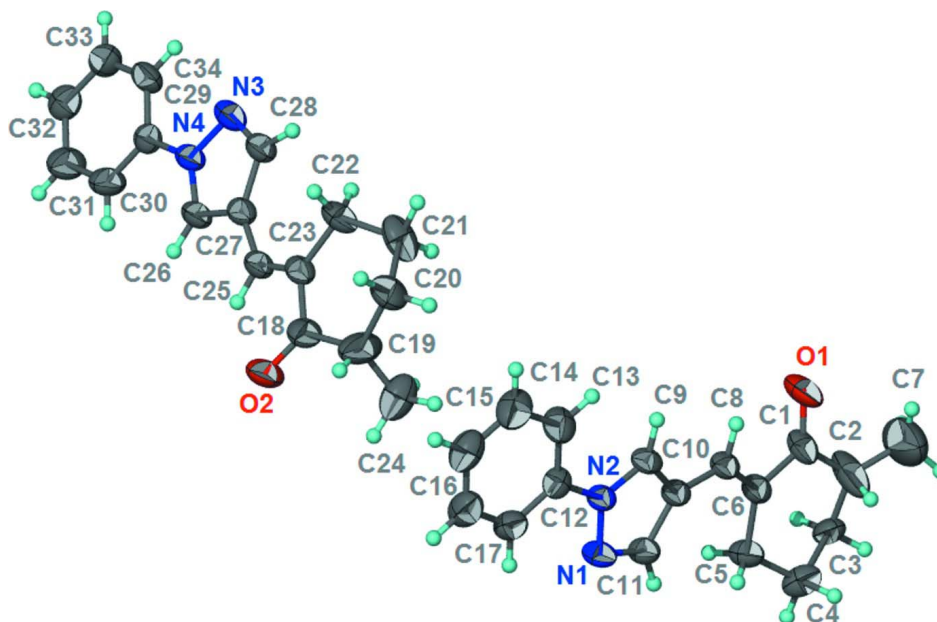
**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of the two independent molecules of $C_{17}H_{18}N_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The minor disorder component is not shown.

## (*E*)-2-Methyl-6-[(1-phenyl-1*H*-pyrazol-4- yl)methylidene]cyclohexanone

*Crystal data*

| | |
|---|---|
| $C_{17}H_{18}N_2O$ | $Z = 4$ |
| $M_r = 266.33$ | $F(000) = 568$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.283$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 6.1152$ (8) Å | Cell parameters from 2004 reflections |
| $b = 10.3757$ (13) Å | $\theta = 3.5$–$23.0°$ |
| $c = 22.734$ (3) Å | $\mu = 0.08$ mm$^{-1}$ |
| $\alpha = 77.542$ (2)° | $T = 100$ K |
| $\beta = 89.667$ (2)° | Block, colorless |
| $\gamma = 78.510$ (2)° | $0.20 \times 0.20 \times 0.05$ mm |
| $V = 1379.2$ (3) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX diffractometer | 3235 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.051$ |
| Graphite monochromator | $\theta_{max} = 25.1°$, $\theta_{min} = 0.9°$ |
| $\omega$ scans | $h = -7 \rightarrow 7$ |
| 14492 measured reflections | $k = -12 \rightarrow 12$ |
| 4890 independent reflections | $l = -27 \rightarrow 27$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $S = 1.03$ |
| Least-squares matrix: full | 4890 reflections |
| $R[F^2 > 2\sigma(F^2)] = 0.062$ | 408 parameters |
| $wR(F^2) = 0.172$ | 47 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

$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 1.0076P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.40$ e Å$^{-3}$

$\Delta\rho_{min} = -0.27$ e Å$^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2$\theta$)]$^{-1/4}$

Extinction coefficient: 0.0074 (17)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

|  | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | 0.7546 (4) | 1.1287 (2) | 0.43105 (11) | 0.0512 (7) | |
| O2 | 0.7707 (4) | 0.4267 (2) | 0.91788 (10) | 0.0458 (6) | |
| N1 | 0.7423 (4) | 0.5112 (2) | 0.55321 (12) | 0.0347 (6) | |
| N2 | 0.9381 (4) | 0.5341 (2) | 0.57342 (10) | 0.0286 (6) | |
| N3 | 0.7798 (4) | 0.9225 (3) | 1.04505 (12) | 0.0384 (7) | |
| N4 | 0.5931 (4) | 0.8732 (2) | 1.06408 (11) | 0.0332 (6) | |
| C1 | 0.6219 (5) | 1.0760 (3) | 0.40999 (14) | 0.0405 (8) | |
| C2 | 0.4788 (6) | 1.1553 (4) | 0.35595 (19) | 0.0722 (14) | |
| H1 | 0.5484 | 1.0941 | 0.3295 | 0.087* | 0.684 (10) |
| H1′ | 0.3958 | 1.2398 | 0.3667 | 0.087* | 0.316 (10) |
| C5 | 0.3955 (5) | 0.8869 (3) | 0.41931 (14) | 0.0389 (8) | |
| H5A | 0.2891 | 0.8917 | 0.4522 | 0.047* | 0.684 (10) |
| H5B | 0.4408 | 0.7910 | 0.4171 | 0.047* | 0.684 (10) |
| H5C | 0.4396 | 0.8219 | 0.3931 | 0.047* | 0.316 (10) |
| H5D | 0.3372 | 0.8386 | 0.4566 | 0.047* | 0.316 (10) |
| C6 | 0.6001 (5) | 0.9351 (3) | 0.43570 (14) | 0.0330 (7) | |
| C8 | 0.7639 (5) | 0.8587 (3) | 0.47404 (13) | 0.0322 (7) | |
| H8 | 0.8830 | 0.9015 | 0.4800 | 0.039* | |
| C9 | 0.9686 (5) | 0.6585 (3) | 0.54691 (13) | 0.0304 (7) | |
| H9 | 1.0927 | 0.6963 | 0.5540 | 0.036* | |
| C10 | 0.7884 (5) | 0.7216 (3) | 0.50772 (13) | 0.0297 (7) | |
| C11 | 0.6536 (5) | 0.6233 (3) | 0.51371 (13) | 0.0331 (7) | |
| H11 | 0.5159 | 0.6363 | 0.4920 | 0.040* | |
| C12 | 1.0725 (5) | 0.4378 (3) | 0.61952 (13) | 0.0309 (7) | |
| C13 | 1.2695 (5) | 0.4635 (4) | 0.64024 (15) | 0.0450 (9) | |
| H13 | 1.3176 | 0.5441 | 0.6222 | 0.054* | |
| C14 | 1.3950 (6) | 0.3724 (4) | 0.68696 (16) | 0.0514 (9) | |
| H14 | 1.5289 | 0.3908 | 0.7011 | 0.062* | |
| C15 | 1.3274 (6) | 0.2545 (4) | 0.71336 (16) | 0.0493 (9) | |
| H15 | 1.4127 | 0.1923 | 0.7460 | 0.059* | |
| C16 | 1.1338 (6) | 0.2282 (3) | 0.69159 (15) | 0.0472 (9) | |
| H16 | 1.0883 | 0.1464 | 0.7090 | 0.057* | |
| C17 | 1.0047 (5) | 0.3195 (3) | 0.64472 (14) | 0.0381 (8) | |
| H17 | 0.8717 | 0.3007 | 0.6302 | 0.046* | |
| C18 | 0.8985 (5) | 0.5011 (3) | 0.89857 (14) | 0.0402 (8) | |
| C19 | 1.0294 (6) | 0.4779 (5) | 0.84412 (18) | 0.0658 (12) | |
| H19 | 1.0803 | 0.3777 | 0.8544 | 0.079* | 0.636 (10) |

| | | | | | |
|---|---|---|---|---|---|
| H19′ | 1.1488 | 0.3974 | 0.8610 | 0.079* | 0.364 (10) |
| C22 | 1.1215 (5) | 0.6813 (3) | 0.91155 (15) | 0.0417 (8) | |
| H22A | 1.2378 | 0.6422 | 0.9441 | 0.050* | |
| H22B | 1.0741 | 0.7785 | 0.9110 | 0.050* | |
| C23 | 0.9246 (5) | 0.6151 (3) | 0.92600 (14) | 0.0344 (7) | |
| C25 | 0.7668 (5) | 0.6508 (3) | 0.96382 (13) | 0.0348 (7) | |
| H25 | 0.6523 | 0.6000 | 0.9684 | 0.042* | |
| C26 | 0.5680 (5) | 0.7727 (3) | 1.03684 (14) | 0.0349 (7) | |
| H26 | 0.4508 | 0.7239 | 1.0430 | 0.042* | |
| C27 | 0.7411 (5) | 0.7534 (3) | 0.99881 (13) | 0.0334 (7) | |
| C28 | 0.8673 (5) | 0.8510 (3) | 1.00599 (14) | 0.0369 (7) | |
| H28 | 0.9992 | 0.8632 | 0.9851 | 0.044* | |
| C29 | 0.4592 (5) | 0.9232 (3) | 1.10852 (13) | 0.0326 (7) | |
| C30 | 0.2761 (5) | 0.8690 (3) | 1.12765 (15) | 0.0439 (8) | |
| H30 | 0.2409 | 0.7980 | 1.1116 | 0.053* | |
| C31 | 0.1439 (6) | 0.9192 (4) | 1.17057 (16) | 0.0490 (9) | |
| H31 | 0.0161 | 0.8831 | 1.1833 | 0.059* | |
| C32 | 0.1950 (6) | 1.0208 (3) | 1.19508 (15) | 0.0415 (8) | |
| H32 | 0.1048 | 1.0538 | 1.2249 | 0.050* | |
| C33 | 0.3778 (6) | 1.0733 (3) | 1.17562 (15) | 0.0444 (8) | |
| H33 | 0.4132 | 1.1438 | 1.1920 | 0.053* | |
| C34 | 0.5120 (6) | 1.0256 (3) | 1.13250 (15) | 0.0414 (8) | |
| H34 | 0.6388 | 1.0627 | 1.1195 | 0.050* | |
| C3 | 0.2541 (10) | 1.1187 (6) | 0.3523 (3) | 0.0381 (17) | 0.684 (10) |
| H3A | 0.1785 | 1.1671 | 0.3131 | 0.046* | 0.684 (10) |
| H3B | 0.1613 | 1.1486 | 0.3846 | 0.046* | 0.684 (10) |
| C4 | 0.2715 (9) | 0.9670 (5) | 0.3587 (3) | 0.0426 (16) | 0.684 (10) |
| H4A | 0.3534 | 0.9378 | 0.3246 | 0.051* | 0.684 (10) |
| H4B | 0.1198 | 0.9474 | 0.3574 | 0.051* | 0.684 (10) |
| C7 | 0.5375 (19) | 1.2686 (11) | 0.3194 (4) | 0.135 (5) | 0.684 (10) |
| H7A | 0.6956 | 1.2674 | 0.3270 | 0.202* | 0.684 (10) |
| H7B | 0.4459 | 1.3503 | 0.3285 | 0.202* | 0.684 (10) |
| H7C | 0.5123 | 1.2677 | 0.2769 | 0.202* | 0.684 (10) |
| C20 | 1.2497 (11) | 0.5233 (8) | 0.8404 (4) | 0.054 (2) | 0.636 (10) |
| H20A | 1.3110 | 0.5201 | 0.8002 | 0.065* | 0.636 (10) |
| H20B | 1.3569 | 0.4605 | 0.8711 | 0.065* | 0.636 (10) |
| C21 | 1.2252 (13) | 0.6658 (7) | 0.8506 (3) | 0.054 (2) | 0.636 (10) |
| H21A | 1.1300 | 0.7296 | 0.8175 | 0.065* | 0.636 (10) |
| H21B | 1.3741 | 0.6900 | 0.8493 | 0.065* | 0.636 (10) |
| C24 | 0.8901 (11) | 0.4964 (9) | 0.7912 (3) | 0.055 (2) | 0.636 (10) |
| H24A | 0.7938 | 0.4300 | 0.7980 | 0.083* | 0.636 (10) |
| H24B | 0.9839 | 0.4844 | 0.7569 | 0.083* | 0.636 (10) |
| H24C | 0.7975 | 0.5875 | 0.7826 | 0.083* | 0.636 (10) |
| C3′ | 0.304 (2) | 1.0840 (16) | 0.3361 (5) | 0.042 (4) | 0.316 (10) |
| H3′A | 0.1804 | 1.1527 | 0.3134 | 0.050* | 0.316 (10) |
| H3′B | 0.3736 | 1.0270 | 0.3085 | 0.050* | 0.316 (10) |
| C4′ | 0.2123 (16) | 0.9985 (11) | 0.3874 (6) | 0.045 (4)* | 0.316 (10) |
| H4′A | 0.0937 | 0.9600 | 0.3723 | 0.054* | 0.316 (10) |

| | | | | | |
|---|---|---|---|---|---|
| H4′B | 0.1455 | 1.0540 | 0.4159 | 0.054* | 0.316 (10) |
| C7′ | 0.6175 (13) | 1.1961 (9) | 0.3041 (3) | 0.021 (2)* | 0.316 (10) |
| H7′A | 0.7578 | 1.2121 | 0.3189 | 0.031* | 0.316 (10) |
| H7′B | 0.5363 | 1.2789 | 0.2775 | 0.031* | 0.316 (10) |
| H7′C | 0.6501 | 1.1242 | 0.2816 | 0.031* | 0.316 (10) |
| C20′ | 1.1615 (18) | 0.5886 (11) | 0.8212 (4) | 0.039 (3) | 0.364 (10) |
| H20C | 1.2677 | 0.5612 | 0.7910 | 0.047* | 0.364 (10) |
| H20D | 1.0581 | 0.6731 | 0.8017 | 0.047* | 0.364 (10) |
| C21′ | 1.2891 (14) | 0.6119 (12) | 0.8744 (5) | 0.037 (3) | 0.364 (10) |
| H21C | 1.3702 | 0.5248 | 0.8989 | 0.044* | 0.364 (10) |
| H21D | 1.3989 | 0.6686 | 0.8596 | 0.044* | 0.364 (10) |
| C24′ | 0.916 (3) | 0.4338 (18) | 0.7979 (5) | 0.087 (6) | 0.364 (10) |
| H24D | 0.8396 | 0.3621 | 0.8171 | 0.130* | 0.364 (10) |
| H24E | 1.0261 | 0.3997 | 0.7705 | 0.130* | 0.364 (10) |
| H24F | 0.8067 | 0.5101 | 0.7750 | 0.130* | 0.364 (10) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0430 (14) | 0.0421 (14) | 0.0690 (17) | −0.0242 (11) | −0.0029 (12) | 0.0011 (12) |
| O2 | 0.0393 (13) | 0.0510 (14) | 0.0572 (15) | −0.0256 (11) | 0.0085 (11) | −0.0189 (12) |
| N1 | 0.0263 (13) | 0.0344 (14) | 0.0452 (16) | −0.0107 (11) | −0.0039 (12) | −0.0084 (12) |
| N2 | 0.0234 (12) | 0.0321 (13) | 0.0324 (14) | −0.0090 (10) | 0.0019 (10) | −0.0084 (11) |
| N3 | 0.0327 (14) | 0.0367 (15) | 0.0458 (16) | −0.0152 (12) | 0.0002 (12) | −0.0012 (13) |
| N4 | 0.0311 (14) | 0.0322 (14) | 0.0365 (15) | −0.0136 (11) | −0.0047 (11) | −0.0013 (11) |
| C1 | 0.0305 (17) | 0.0417 (19) | 0.047 (2) | −0.0142 (15) | 0.0048 (15) | 0.0015 (16) |
| C2 | 0.044 (2) | 0.076 (3) | 0.081 (3) | −0.031 (2) | −0.013 (2) | 0.034 (2) |
| C5 | 0.0378 (18) | 0.0356 (17) | 0.046 (2) | −0.0123 (14) | −0.0051 (15) | −0.0097 (15) |
| C6 | 0.0293 (16) | 0.0352 (17) | 0.0364 (17) | −0.0115 (13) | 0.0055 (14) | −0.0078 (14) |
| C8 | 0.0284 (16) | 0.0372 (17) | 0.0351 (17) | −0.0138 (13) | 0.0071 (13) | −0.0105 (14) |
| C9 | 0.0228 (15) | 0.0344 (16) | 0.0384 (17) | −0.0111 (12) | 0.0064 (13) | −0.0128 (14) |
| C10 | 0.0265 (15) | 0.0330 (16) | 0.0328 (16) | −0.0095 (13) | 0.0048 (13) | −0.0109 (13) |
| C11 | 0.0299 (16) | 0.0343 (17) | 0.0367 (18) | −0.0098 (13) | −0.0032 (14) | −0.0082 (14) |
| C12 | 0.0251 (15) | 0.0367 (17) | 0.0297 (16) | −0.0021 (13) | 0.0039 (13) | −0.0085 (13) |
| C13 | 0.0300 (17) | 0.060 (2) | 0.043 (2) | −0.0142 (16) | 0.0006 (15) | −0.0015 (17) |
| C14 | 0.0299 (18) | 0.076 (3) | 0.044 (2) | −0.0071 (18) | −0.0025 (16) | −0.0081 (19) |
| C15 | 0.048 (2) | 0.056 (2) | 0.0375 (19) | 0.0061 (18) | −0.0018 (17) | −0.0101 (17) |
| C16 | 0.063 (2) | 0.0353 (19) | 0.041 (2) | −0.0049 (17) | −0.0053 (18) | −0.0072 (15) |
| C17 | 0.0412 (18) | 0.0371 (18) | 0.0382 (18) | −0.0083 (14) | −0.0027 (15) | −0.0123 (15) |
| C18 | 0.0291 (17) | 0.052 (2) | 0.044 (2) | −0.0163 (15) | 0.0012 (15) | −0.0128 (16) |
| C19 | 0.049 (2) | 0.112 (4) | 0.061 (3) | −0.045 (2) | 0.019 (2) | −0.047 (2) |
| C22 | 0.0311 (17) | 0.0359 (18) | 0.058 (2) | −0.0143 (14) | 0.0004 (16) | −0.0026 (16) |
| C23 | 0.0288 (16) | 0.0362 (17) | 0.0364 (18) | −0.0121 (13) | −0.0058 (14) | 0.0012 (14) |
| C25 | 0.0316 (16) | 0.0374 (17) | 0.0359 (17) | −0.0163 (14) | −0.0037 (14) | −0.0005 (14) |
| C26 | 0.0327 (17) | 0.0336 (17) | 0.0389 (18) | −0.0160 (14) | −0.0045 (14) | −0.0004 (14) |
| C27 | 0.0311 (16) | 0.0343 (17) | 0.0337 (17) | −0.0123 (13) | −0.0056 (13) | 0.0000 (14) |
| C28 | 0.0337 (17) | 0.0390 (18) | 0.0394 (18) | −0.0163 (14) | 0.0000 (14) | −0.0034 (15) |
| C29 | 0.0333 (16) | 0.0320 (16) | 0.0299 (16) | −0.0072 (13) | −0.0045 (13) | −0.0002 (13) |

| | | | | | |
|---|---|---|---|---|---|
| C30 | 0.0383 (18) | 0.052 (2) | 0.049 (2) | −0.0198 (16) | 0.0008 (16) | −0.0180 (17) |
| C31 | 0.0371 (19) | 0.062 (2) | 0.054 (2) | −0.0178 (17) | 0.0030 (17) | −0.0201 (19) |
| C32 | 0.0423 (19) | 0.0391 (18) | 0.0384 (19) | −0.0016 (15) | −0.0043 (15) | −0.0047 (15) |
| C33 | 0.063 (2) | 0.0317 (17) | 0.0393 (19) | −0.0125 (16) | −0.0009 (17) | −0.0074 (15) |
| C34 | 0.049 (2) | 0.0315 (17) | 0.043 (2) | −0.0183 (15) | −0.0006 (16) | 0.0008 (15) |
| C3 | 0.049 (4) | 0.038 (3) | 0.033 (4) | −0.014 (3) | −0.006 (3) | −0.012 (3) |
| C4 | 0.044 (3) | 0.038 (3) | 0.044 (3) | 0.003 (2) | −0.011 (3) | −0.015 (2) |
| C7 | 0.197 (11) | 0.124 (9) | 0.086 (6) | −0.104 (9) | −0.040 (7) | 0.036 (6) |
| C20 | 0.042 (4) | 0.068 (5) | 0.058 (5) | −0.030 (4) | 0.006 (3) | −0.010 (4) |
| C21 | 0.034 (4) | 0.049 (4) | 0.069 (5) | −0.005 (3) | 0.018 (4) | 0.003 (4) |
| C24 | 0.051 (4) | 0.065 (5) | 0.042 (4) | 0.010 (3) | 0.002 (3) | −0.014 (3) |
| C3′ | 0.059 (7) | 0.040 (7) | 0.034 (7) | −0.012 (6) | −0.011 (5) | −0.024 (5) |
| C20′ | 0.041 (6) | 0.038 (6) | 0.038 (5) | −0.006 (5) | 0.010 (4) | −0.008 (4) |
| C21′ | 0.025 (5) | 0.030 (6) | 0.055 (6) | −0.009 (4) | 0.003 (4) | −0.006 (5) |
| C24′ | 0.112 (10) | 0.088 (10) | 0.091 (9) | −0.064 (8) | 0.022 (7) | −0.047 (7) |

*Geometric parameters (Å, º)*

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|---|---|---|---|
| O1—C1 | 1.217 (4) | C22—C21 | 1.547 (6) |
| O2—C18 | 1.220 (4) | C22—H22A | 0.9900 |
| N1—C11 | 1.326 (4) | C22—H22B | 0.9900 |
| N1—N2 | 1.367 (3) | C23—C25 | 1.339 (4) |
| N2—C9 | 1.350 (4) | C25—C27 | 1.445 (4) |
| N2—C12 | 1.415 (4) | C25—H25 | 0.9500 |
| N3—C28 | 1.322 (4) | C26—C27 | 1.374 (4) |
| N3—N4 | 1.373 (3) | C26—H26 | 0.9500 |
| N4—C26 | 1.355 (4) | C27—C28 | 1.426 (4) |
| N4—C29 | 1.417 (4) | C28—H28 | 0.9500 |
| C1—C6 | 1.485 (4) | C29—C30 | 1.378 (4) |
| C1—C2 | 1.497 (5) | C29—C34 | 1.386 (4) |
| C2—C7 | 1.395 (6) | C30—C31 | 1.385 (5) |
| C2—C7′ | 1.485 (7) | C30—H30 | 0.9500 |
| C2—C3 | 1.504 (6) | C31—C32 | 1.381 (5) |
| C2—C3′ | 1.533 (8) | C31—H31 | 0.9500 |
| C2—H1 | 1.0000 | C32—C33 | 1.369 (5) |
| C2—H1′ | 1.0000 | C32—H32 | 0.9500 |
| C5—C4′ | 1.501 (8) | C33—C34 | 1.385 (5) |
| C5—C6 | 1.513 (4) | C33—H33 | 0.9500 |
| C5—C4 | 1.559 (5) | C34—H34 | 0.9500 |
| C5—H5A | 0.9900 | C3—C4 | 1.532 (6) |
| C5—H5B | 0.9900 | C3—H3A | 0.9900 |
| C5—H5C | 0.9900 | C3—H3B | 0.9900 |
| C5—H5D | 0.9900 | C4—H4A | 0.9900 |
| C6—C8 | 1.337 (4) | C4—H4B | 0.9900 |
| C8—C10 | 1.442 (4) | C7—H7A | 0.9800 |
| C8—H8 | 0.9500 | C7—H7B | 0.9800 |
| C9—C10 | 1.381 (4) | C7—H7C | 0.9800 |
| C9—H9 | 0.9500 | C20—C21 | 1.524 (8) |

| | | | |
|---|---|---|---|
| C10—C11 | 1.418 (4) | C20—H20A | 0.9900 |
| C11—H11 | 0.9500 | C20—H20B | 0.9900 |
| C12—C17 | 1.380 (4) | C21—H21A | 0.9900 |
| C12—C13 | 1.390 (4) | C21—H21B | 0.9900 |
| C13—C14 | 1.378 (5) | C24—H24A | 0.9800 |
| C13—H13 | 0.9500 | C24—H24B | 0.9800 |
| C14—C15 | 1.382 (5) | C24—H24C | 0.9800 |
| C14—H14 | 0.9500 | C3′—C4′ | 1.486 (9) |
| C15—C16 | 1.384 (5) | C3′—H3′A | 0.9900 |
| C15—H15 | 0.9500 | C3′—H3′B | 0.9900 |
| C16—C17 | 1.390 (4) | C4′—H4′A | 0.9900 |
| C16—H16 | 0.9500 | C4′—H4′B | 0.9900 |
| C17—H17 | 0.9500 | C7′—H7′A | 0.9800 |
| C18—C23 | 1.486 (4) | C7′—H7′B | 0.9800 |
| C18—C19 | 1.507 (5) | C7′—H7′C | 0.9800 |
| C19—C24 | 1.433 (7) | C20′—C21′ | 1.530 (9) |
| C19—C24′ | 1.462 (8) | C20′—H20C | 0.9900 |
| C19—C20 | 1.509 (6) | C20′—H20D | 0.9900 |
| C19—C20′ | 1.533 (8) | C21′—H21C | 0.9900 |
| C19—H19 | 1.0000 | C21′—H21D | 0.9900 |
| C19—H19′ | 1.0000 | C24′—H24D | 0.9800 |
| C22—C21′ | 1.497 (8) | C24′—H24E | 0.9800 |
| C22—C23 | 1.502 (4) | C24′—H24F | 0.9800 |
| | | | |
| C11—N1—N2 | 105.1 (2) | C26—C27—C25 | 122.4 (3) |
| C9—N2—N1 | 111.1 (2) | C28—C27—C25 | 134.1 (3) |
| C9—N2—C12 | 127.9 (2) | N3—C28—C27 | 112.2 (3) |
| N1—N2—C12 | 120.9 (2) | N3—C28—H28 | 123.9 |
| C28—N3—N4 | 104.9 (2) | C27—C28—H28 | 123.9 |
| C26—N4—N3 | 111.1 (3) | C30—C29—C34 | 120.4 (3) |
| C26—N4—C29 | 128.0 (2) | C30—C29—N4 | 119.4 (3) |
| N3—N4—C29 | 120.9 (2) | C34—C29—N4 | 120.3 (3) |
| O1—C1—C6 | 122.0 (3) | C29—C30—C31 | 119.3 (3) |
| O1—C1—C2 | 118.8 (3) | C29—C30—H30 | 120.4 |
| C6—C1—C2 | 119.1 (3) | C31—C30—H30 | 120.4 |
| C7—C2—C1 | 120.9 (4) | C32—C31—C30 | 121.1 (3) |
| C7′—C2—C1 | 111.0 (4) | C32—C31—H31 | 119.5 |
| C1—C2—C3 | 114.4 (3) | C30—C31—H31 | 119.5 |
| C7′—C2—C3′ | 109.2 (6) | C33—C32—C31 | 118.9 (3) |
| C1—C2—C3′ | 114.7 (6) | C33—C32—H32 | 120.6 |
| C7′—C2—H1′ | 107.2 | C31—C32—H32 | 120.6 |
| C1—C2—H1′ | 107.2 | C32—C33—C34 | 121.3 (3) |
| C3′—C2—H1′ | 107.2 | C32—C33—H33 | 119.4 |
| C4′—C5—C6 | 113.6 (5) | C34—C33—H33 | 119.4 |
| C6—C5—C4 | 115.3 (3) | C29—C34—C33 | 119.2 (3) |
| C6—C5—H5A | 108.4 | C29—C34—H34 | 120.4 |
| C4—C5—H5A | 108.4 | C33—C34—H34 | 120.4 |
| C6—C5—H5B | 108.4 | C2—C3—C4 | 112.4 (5) |

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|---|---|---|---|
| C4—C5—H5B | 108.4 | C2—C3—H3A | 109.1 |
| H5A—C5—H5B | 107.5 | C4—C3—H3A | 109.1 |
| C4′—C5—H5C | 108.8 | C2—C3—H3B | 109.1 |
| C6—C5—H5C | 108.8 | C4—C3—H3B | 109.1 |
| C4′—C5—H5D | 108.8 | H3A—C3—H3B | 107.9 |
| C6—C5—H5D | 108.8 | C3—C4—C5 | 110.6 (4) |
| H5C—C5—H5D | 107.7 | C3—C4—H4A | 109.5 |
| C8—C6—C1 | 116.7 (3) | C5—C4—H4A | 109.5 |
| C8—C6—C5 | 123.7 (3) | C3—C4—H4B | 109.5 |
| C1—C6—C5 | 119.6 (3) | C5—C4—H4B | 109.5 |
| C6—C8—C10 | 130.2 (3) | H4A—C4—H4B | 108.1 |
| C6—C8—H8 | 114.9 | C2—C7—H7A | 109.5 |
| C10—C8—H8 | 114.9 | C2—C7—H7B | 109.5 |
| N2—C9—C10 | 108.3 (2) | H7A—C7—H7B | 109.5 |
| N2—C9—H9 | 125.8 | C2—C7—H7C | 109.5 |
| C10—C9—H9 | 125.8 | H7A—C7—H7C | 109.5 |
| C9—C10—C11 | 103.4 (3) | H7B—C7—H7C | 109.5 |
| C9—C10—C8 | 122.5 (3) | C19—C20—C21 | 111.9 (6) |
| C11—C10—C8 | 134.0 (3) | C19—C20—H20A | 109.2 |
| N1—C11—C10 | 112.1 (3) | C21—C20—H20A | 109.2 |
| N1—C11—H11 | 124.0 | C19—C20—H20B | 109.2 |
| C10—C11—H11 | 124.0 | C21—C20—H20B | 109.2 |
| C17—C12—C13 | 120.1 (3) | H20A—C20—H20B | 107.9 |
| C17—C12—N2 | 119.7 (3) | C20—C21—C22 | 112.7 (5) |
| C13—C12—N2 | 120.2 (3) | C20—C21—H21A | 109.1 |
| C14—C13—C12 | 120.1 (3) | C22—C21—H21A | 109.1 |
| C14—C13—H13 | 119.9 | C20—C21—H21B | 109.1 |
| C12—C13—H13 | 119.9 | C22—C21—H21B | 109.1 |
| C13—C14—C15 | 120.5 (3) | H21A—C21—H21B | 107.8 |
| C13—C14—H14 | 119.8 | C19—C24—H24A | 109.5 |
| C15—C14—H14 | 119.8 | C19—C24—H24B | 109.5 |
| C14—C15—C16 | 119.1 (3) | H24A—C24—H24B | 109.5 |
| C14—C15—H15 | 120.5 | C19—C24—H24C | 109.5 |
| C16—C15—H15 | 120.5 | H24A—C24—H24C | 109.5 |
| C15—C16—C17 | 121.1 (3) | H24B—C24—H24C | 109.5 |
| C15—C16—H16 | 119.4 | C4′—C3′—C2 | 113.2 (9) |
| C17—C16—H16 | 119.4 | C4′—C3′—H3′A | 108.9 |
| C12—C17—C16 | 119.1 (3) | C2—C3′—H3′A | 108.9 |
| C12—C17—H17 | 120.5 | C4′—C3′—H3′B | 108.9 |
| C16—C17—H17 | 120.5 | C2—C3′—H3′B | 108.9 |
| O2—C18—C23 | 122.2 (3) | H3′A—C3′—H3′B | 107.8 |
| O2—C18—C19 | 118.5 (3) | C3′—C4′—C5 | 109.6 (10) |
| C23—C18—C19 | 119.2 (3) | C3′—C4′—H4′A | 109.7 |
| C24—C19—C20 | 121.1 (5) | C5—C4′—H4′A | 109.7 |
| C24—C19—C18 | 113.0 (4) | C3′—C4′—H4′B | 109.7 |
| C24′—C19—C18 | 116.8 (6) | C5—C4′—H4′B | 109.7 |
| C20—C19—C18 | 114.6 (4) | H4′A—C4′—H4′B | 108.2 |
| C24′—C19—C20′ | 116.0 (6) | C2—C7′—H7′A | 109.5 |

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|---|---|---|---|
| C18—C19—C20′ | 112.2 (5) | C2—C7′—H7′B | 109.5 |
| C24—C19—H19 | 101.3 | H7′A—C7′—H7′B | 109.5 |
| C20—C19—H19 | 101.3 | C2—C7′—H7′C | 109.5 |
| C18—C19—H19 | 101.3 | H7′A—C7′—H7′C | 109.5 |
| C24′—C19—H19′ | 103.1 | H7′B—C7′—H7′C | 109.5 |
| C18—C19—H19′ | 103.1 | C21′—C20′—C19 | 109.2 (8) |
| C20′—C19—H19′ | 103.1 | C21′—C20′—H20C | 109.8 |
| C21′—C22—C23 | 113.7 (5) | C19—C20′—H20C | 109.8 |
| C23—C22—C21 | 114.2 (4) | C21′—C20′—H20D | 109.8 |
| C23—C22—H22A | 108.7 | C19—C20′—H20D | 109.8 |
| C21—C22—H22A | 108.7 | H20C—C20′—H20D | 108.3 |
| C23—C22—H22B | 108.7 | C22—C21′—C20′ | 107.5 (8) |
| C21—C22—H22B | 108.7 | C22—C21′—H21C | 110.2 |
| H22A—C22—H22B | 107.6 | C20′—C21′—H21C | 110.2 |
| C25—C23—C18 | 115.9 (3) | C22—C21′—H21D | 110.2 |
| C25—C23—C22 | 124.0 (3) | C20′—C21′—H21D | 110.2 |
| C18—C23—C22 | 120.1 (3) | H21C—C21′—H21D | 108.5 |
| C23—C25—C27 | 131.0 (3) | C19—C24′—H24D | 109.5 |
| C23—C25—H25 | 114.5 | C19—C24′—H24E | 109.5 |
| C27—C25—H25 | 114.5 | H24D—C24′—H24E | 109.5 |
| N4—C26—C27 | 108.4 (3) | C19—C24′—H24F | 109.5 |
| N4—C26—H26 | 125.8 | H24D—C24′—H24F | 109.5 |
| C27—C26—H26 | 125.8 | H24E—C24′—H24F | 109.5 |
| C26—C27—C28 | 103.5 (3) | | |
| | | | |
| C11—N1—N2—C9 | 0.2 (3) | C21′—C22—C23—C25 | 172.6 (5) |
| C11—N1—N2—C12 | 176.0 (2) | C21—C22—C23—C25 | −156.8 (4) |
| C28—N3—N4—C26 | 0.0 (3) | C21′—C22—C23—C18 | −6.3 (6) |
| C28—N3—N4—C29 | 177.7 (3) | C21—C22—C23—C18 | 24.3 (5) |
| O1—C1—C2—C7 | 19.2 (10) | C18—C23—C25—C27 | 178.7 (3) |
| C6—C1—C2—C7 | −160.0 (8) | C22—C23—C25—C27 | −0.1 (5) |
| O1—C1—C2—C7′ | 60.4 (6) | N3—N4—C26—C27 | 0.2 (3) |
| C6—C1—C2—C7′ | −118.8 (5) | C29—N4—C26—C27 | −177.2 (3) |
| O1—C1—C2—C3 | −149.8 (4) | N4—C26—C27—C28 | −0.3 (3) |
| C6—C1—C2—C3 | 31.0 (6) | N4—C26—C27—C25 | 178.5 (3) |
| O1—C1—C2—C3′ | −175.3 (7) | C23—C25—C27—C26 | −178.2 (3) |
| C6—C1—C2—C3′ | 5.6 (8) | C23—C25—C27—C28 | 0.1 (6) |
| O1—C1—C6—C8 | −14.5 (5) | N4—N3—C28—C27 | −0.2 (3) |
| C2—C1—C6—C8 | 164.6 (3) | C26—C27—C28—N3 | 0.3 (3) |
| O1—C1—C6—C5 | 163.7 (3) | C25—C27—C28—N3 | −178.2 (3) |
| C2—C1—C6—C5 | −17.2 (5) | C26—N4—C29—C30 | −1.2 (4) |
| C4′—C5—C6—C8 | 166.4 (6) | N3—N4—C29—C30 | −178.4 (3) |
| C4—C5—C6—C8 | −158.6 (4) | C26—N4—C29—C34 | 178.7 (3) |
| C4′—C5—C6—C1 | −11.6 (7) | N3—N4—C29—C34 | 1.6 (4) |
| C4—C5—C6—C1 | 23.4 (5) | C34—C29—C30—C31 | 0.8 (5) |
| C1—C6—C8—C10 | 177.7 (3) | N4—C29—C30—C31 | −179.2 (3) |
| C5—C6—C8—C10 | −0.4 (5) | C29—C30—C31—C32 | −1.1 (5) |
| N1—N2—C9—C10 | 0.0 (3) | C30—C31—C32—C33 | 0.9 (5) |

| | | | |
|---|---|---|---|
| C12—N2—C9—C10 | −175.3 (3) | C31—C32—C33—C34 | −0.5 (5) |
| N2—C9—C10—C11 | −0.3 (3) | C30—C29—C34—C33 | −0.4 (5) |
| N2—C9—C10—C8 | 177.2 (3) | N4—C29—C34—C33 | 179.6 (3) |
| C6—C8—C10—C9 | −178.4 (3) | C32—C33—C34—C29 | 0.3 (5) |
| C6—C8—C10—C11 | −1.8 (6) | C7—C2—C3—C4 | 139.6 (8) |
| N2—N1—C11—C10 | −0.4 (3) | C7′—C2—C3—C4 | 92.8 (7) |
| C9—C10—C11—N1 | 0.5 (3) | C1—C2—C3—C4 | −51.8 (8) |
| C8—C10—C11—N1 | −176.6 (3) | C3′—C2—C3—C4 | 44.3 (15) |
| C9—N2—C12—C17 | 174.6 (3) | C2—C3—C4—C5 | 57.3 (8) |
| N1—N2—C12—C17 | −0.4 (4) | C4′—C5—C4—C3 | 51.7 (8) |
| C9—N2—C12—C13 | −4.0 (4) | C6—C5—C4—C3 | −42.7 (6) |
| N1—N2—C12—C13 | −179.0 (3) | C24—C19—C20—C21 | 92.5 (9) |
| C17—C12—C13—C14 | −1.5 (5) | C24′—C19—C20—C21 | 121.6 (13) |
| N2—C12—C13—C14 | 177.1 (3) | C18—C19—C20—C21 | −48.6 (9) |
| C12—C13—C14—C15 | 0.4 (5) | C20′—C19—C20—C21 | 43.9 (8) |
| C13—C14—C15—C16 | 1.0 (5) | C19—C20—C21—C22 | 56.7 (10) |
| C14—C15—C16—C17 | −1.4 (5) | C21′—C22—C21—C20 | 51.3 (9) |
| C13—C12—C17—C16 | 1.2 (5) | C23—C22—C21—C20 | −44.0 (8) |
| N2—C12—C17—C16 | −177.5 (3) | C7—C2—C3′—C4′ | −160.3 (10) |
| C15—C16—C17—C12 | 0.3 (5) | C7′—C2—C3′—C4′ | 159.8 (10) |
| O2—C18—C19—C24 | 63.2 (6) | C1—C2—C3′—C4′ | 34.5 (15) |
| C23—C18—C19—C24 | −115.3 (5) | C3—C2—C3′—C4′ | −60.1 (12) |
| O2—C18—C19—C24′ | 36.2 (10) | C2—C3′—C4′—C5 | −63.2 (17) |
| C23—C18—C19—C24′ | −142.4 (9) | C6—C5—C4′—C3′ | 50.6 (13) |
| O2—C18—C19—C20 | −152.5 (5) | C4—C5—C4′—C3′ | −49.8 (8) |
| C23—C18—C19—C20 | 29.0 (6) | C24—C19—C20′—C21′ | 167.5 (9) |
| O2—C18—C19—C20′ | 173.6 (5) | C24′—C19—C20′—C21′ | −174.0 (12) |
| C23—C18—C19—C20′ | −5.0 (7) | C20—C19—C20′—C21′ | −53.1 (8) |
| O2—C18—C23—C25 | −14.5 (5) | C18—C19—C20′—C21′ | 48.2 (10) |
| C19—C18—C23—C25 | 164.0 (3) | C23—C22—C21′—C20′ | 48.9 (10) |
| O2—C18—C23—C22 | 164.5 (3) | C21—C22—C21′—C20′ | −48.4 (8) |
| C19—C18—C23—C22 | −17.1 (5) | C19—C20′—C21′—C22 | −71.6 (12) |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C9—H9···O1[i] | 0.95 | 2.29 | 3.157 (4) | 152 |
| C26—H26···O2[ii] | 0.95 | 2.30 | 3.224 (4) | 164 |
| C30—H30···O2[ii] | 0.95 | 2.57 | 3.506 (4) | 167 |

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