The co-crystal 4,6-diacytelyresorcinol–1-aminopyrene (2/1)

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The structure of the title molecular complex, C_{16}H_{11}N·2C_{10}H_{10}O_{4}, at 150 K has been determined. The molecules form stacks consisting of aggregates with disordered 1-aminopyrene molecule surrounded by two 4,6-diacytelyresorcinol molecules. Neighbouring stacks are linked by hydrogen bonds between the amine H atoms of the 1-aminopyrene molecule with the adjacent carbonyl oxygen atom of the 4,6-diacytelyresorcinol molecule.

1. Chemical context

Co-crystals are crystalline single phase materials made up of molecules of two or more compounds. They are used in a variety of fields, including paper, textiles and the chemical, photographic, and electronic industries (Golbedaghi & Fausto, 2018). However, their main uses are centered in the pharmaceutical industry, where they have been gaining importance in recent years.

Schiff bases are the products of the condensation reaction of aldehydes or ketones with amines. They have multiple uses, for example as pigments and dyes, intermediates in organic synthesis, and as catalysts and polymer stabilizers. They also exhibit a broad range of biological activities. They play an important role in coordination chemistry as they readily form stable complexes with most transition metals (Aazam et al., 2006, 2008, 2010; El-Attar & Aazam, 2021). In the process of the synthesis of such compounds with 4,6-diacytelyresorcinol and 1-aminopyrene as the precursors, a new co-crystal, C_{16}H_{11}N·2C_{10}H_{10}O_{4}, has been obtained.
2. Structural commentary

The formula unit of the title compound consists of two 4,6-diacetylresorcinol molecules and one 1-aminopyrene molecule, which lies on an inversion center. Besides this, this molecule is further disordered so that the amino N atom is distributed over four chemically equivalent positions, at the C11 and C13 atoms, with the occupancies of 0.428 (2) and 0.072 (2) for N1A and N1B, respectively (Fig. 1).

In the 4,6-diacetylresorcinol molecule, the hydroxy groups form intramolecular hydrogen bonds with the oxygen atoms of neighbouring acetyl groups, generating S(6) rings (Table 1).

3. Supramolecular features

In the crystal, the molecules form centrosymmetric aggregates with two molecules of 4,6-diacetylresorcinol positioned on both sides of the 1-aminopyrene molecule (Fig. 2). The mean planes of the aromatic rings of the 4,6-diacetylresorcinol molecules are inclined at 2.91 (10)° to the mean plane of the tetracyclic core of the 1-aminopyrene molecule. A short intercentroid separation Cg1···Cg2 of 3.492 (1) Å is observed in this aggregate, with Cg1 being the centroid of C3–C8 ring of diacetylresorcinol and Cg2 the centroid of one of the aminopyrene rings, C11–C18. These aggregates are packed into stacks by π–π stacking interactions between 4,6-diacetylresorcinol molecules. Neighbouring stacks are linked by hydrogen bonds between the amino H atom of the 1-aminopyrene molecule with the adjacent carbonyl oxygen atom of the 4,6-diacetylresorcinol molecule, thus forming a three-dimensional network (Fig. 3).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.42; May 2021; Groom et al., 2016) gave the structures of the individual components. In the structure of 4,6-diacetylresorcinol (Kokila et al., 1992, refcode VOXPED) the molecule is almost planar, with the oxygen atoms of the acetyl groups deviating by 0.074 (1) and 0.072 (2) Å from the mean plane of the benzene ring. There are intramolecular hydrogen bonds between the oxygen atoms of the acetyl groups and the hydroxy hydrogen atoms. A search for 1-aminopyrene gave two hits for co-crystals composed of 1-aminopyrene molecules with either 7,7′,8,8′-tetracyanoquinodimethane or 3,5-dinitro-

Table 1

|          | D—H   | H···A | D—H   | H···A |
|----------|-------|-------|-------|-------|
| O2—H2    | 0.85 (3) | 1.80 (3) | 2.545 (2) | 146 (2) |
| O3—H3    | 0.84 (2) | 1.80 (2) | 2.542 (2) | 147 (2) |
| N1—H1A   | 0.88 (1) | 2.30 (2) | 3.131 (3) | 156 (4) |
| N1—H1B   | 0.88 (1) | 2.16 (2) | 2.966 (3) | 154 (3) |
| N1B—H1C   | 0.88 (1) | 2.05 (6) | 2.902 (18) | 162 (17) |
| N1B—H1D   | 0.88 (1) | 1.89 (6) | 2.731 (18) | 159 (14) |

Symmetry codes: (i) −x + 1/2, y − 1/2, −z + 1/2; (ii) −x + 1/2, y + 1/2, −z + 1; (iii) x, y, z + 1.
benzoic acid and showed that the NH$_2$ groups can act as H-donors in the intermolecular hydrogen-bonding interactions, as in the title compound. (Mandal et al., 2020, refcode BOYQQY; Mandal et al., 2019, refcode LORBOM).

5. Synthesis and crystallization

A solution of 1-aminopyrene (1 mmol, 0.233 g) dissolved in 10 mL of ethanol was added dropwise to 4,6-diacetylresorcinol (DAR) (0.5 mmol, 0.097 g) dissolved in 10 mL of ethanol, 3 drops of acetic acid were added, and the mixture was stirred for 15 min at room temperature and then for about 3 h under reflux. Yellow fiber-like crystals of the Schiff base ligand were separated. They were filtered off and washed with 4 mL of ethanol, weight = 0.021 g, yield = 7.12%, m.p. = 523 K, m/z = 592.7 (C$_{42}$H$_{28}$N$_{2}$O$_{2}$). The filtrate was left overnight upon which dark-brown rectangular co-crystals were formed, weight = 0.04 g, yield = 19.5%, m.p. = 418 K, m/z = 605.62 (C$_{16}$H$_{11}$N·2C$_{10}$H$_{10}$O$_{4}$). $^1$H NMR (800 MHz, DMSO-d$_6$) δ 12.75 (s, br, –OH), 8.406 (s, 2H, DAR), 8.251 (d, 1H), 7.992 (d, 2H), 7.992 (d, 1H), 7.958 (d, 1H), 87.915 (d, 2H), 7.880 (m, 1H), 7.367 (d, 1H), 6.392 (s, 2H, DAR), 6.314 (s, br, NH2, 2H), 2.661 (s, Me, 12H).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C—N bond distances for the disordered N atom were restrained to be similar. The minor occupancy N1B atom was constrained to have the same ADPs as the C atom to which it is bonded. N—H bond distances were restrained to a target value of 0.88 (2) Å, and the H—N—H and C—N—H bond angles were restrained to be similar to each other. Subject to these conditions the occupancy rates refined to 0.52 (2) and 0.08 (2). O-bound H atoms were refined with Uiso(H) = 1.5Ueq(O). C-bound H atoms were positioned geometrically (C—H = 0.9–0.98 Å) and refined as riding on their parent atoms with Uiso(H) = 1.2–1.5Ueq(C).

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Table 2

Experimental details.

| Crystal data | Chemical formula | C$_{16}$H$_{11}$N·2C$_{10}$H$_{10}$O$_{4}$ |
|--------------|-----------------|----------------------------------------|
| M, g | 605.62 |
| Crystal system, space group | Monoclinic, C2/c |
| Temperature (K) | 150 |
| $\beta$ (Å) | 113.758 (4) |
| $V$ (Å$^3$) | 2840.9 (4) |
| Z | 4 |
| Radiation type | Mo Kα |
| $\mu$ (mm$^{-1}$) | 0.10 |
| Crystal size (mm) | 0.45 × 0.26 × 0.22 |

Data collection

Diffractometer | Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD) |
Absorption correction | Multi-scan (SADABS; Krause et al., 2015) |
$T_{	ext{max}}$, $T_{	ext{min}}$ | 0.673, 0.747 |
No. of measured, independent and observed [I > 2σ(I)] reflections | 33773, 4316, 3741 |
$R_{	ext{int}}$, $(\sin \theta/\lambda)_{\text{max}}$ (Å$^{-1}$) | 0.046, 0.714 |

Refinement

$R[F^2 > 2\sigma(F^2)]$, wR(F$^2$), $S$ | 0.061, 0.153, 1.15 |
No. of reflections | 4316 |
No. of parameters | 231 |
No. of restraints | 13 |
H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
$\Delta$R$_{\text{free}}$, $\Delta$ρ$_{\text{max}}$ (e Å$^{-3}$) | 0.35, −0.26 |

Computer programs: APEX4 and SAINT (Bruker, 2021), SHELXT (Sheldrick, 2015a), SHELXL2019/2 (Sheldrick, 2015b), ShelXle (Hübschle et al., 2011) and publCIF (Westrip, 2010).
supporting information

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The co-crystal 4,6-diacetylresorcinol–1-aminopyrene (2/1)

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Computing details

Data collection: APEX4 (Bruker, 2021); cell refinement: SAINT (Bruker, 2021); data reduction: SAINT (Bruker, 2021); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2019/2 (Sheldrick, 2015b), ShelXle (Hübschle et al., 2011); software used to prepare material for publication: publCIF (Westrip, 2010).

4,6-Diacetylresorcinol–1-aminopyrene (2/1)

Crystal data

C_{16}H_{11}N·2C_{10}H_{10}O_{4}  

Mr = 605.62

Monoclinic, C2/c

a = 18.7222 (14) Å

b = 9.7870 (6) Å

c = 16.9398 (15) Å

β = 113.758 (4)°

V = 2840.9 (4) Å³

Z = 4

F(000) = 1272

D_x = 1.416 Mg m⁻³

Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 9850 reflections

θ = 2.4–33.2°

µ = 0.10 mm⁻¹

T = 150 K

Block, yellow

0.45 × 0.26 × 0.22 mm

Data collection

Bruker AXS D8 Quest
diffractometer with PhotonII charge-integrating pixel array detector (CPAD)

Radiation source: fine focus sealed tube X-ray source

Triumph curved graphite crystal monochromator

Detector resolution: 7.4074 pixels mm⁻¹

ω and phi scans

Absorption correction: multi-scan (SADABS, Krause et al., 2015)

T_min = 0.673, T_max = 0.747

33773 measured reflections

4316 independent reflections

3741 reflections with I > 2σ(I)

R_int = 0.046

θ_max = 30.5°, θ_min = 2.5°

h = −26→26

k = −13→13

l = −24→24

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.061

wR(F²) = 0.153

S = 1.15

4316 reflections

231 parameters

13 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ(F²) + (0.0356P)^2 + 4.9256P]

where P = (F² + 2Ḟc²)/3
(Δ/σ)_{max} < 0.001
Δρ_{max} = 0.35 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. The single nitrogen atom is disordered over four chemically equivalent positions (each two are also crystallographically equivalent, by inversion). The C-N bond distances were restrained to be similar. The minor N atom was constrained to have the same ADP as the C atom it is bonded to. The N-H bond distances were restrained to a target value of 0.88 (2) Ångstrom, and the H-N-H and C-N-H bond angles were each restrained to be similar to each other. Subject to these conditions the occupancy rates refined to two times 0.428 (2) and two times 0.072 (2).

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

|     | x        | y        | z        | U_{iso}/U_{eq} | Occ. (<1) |
|-----|----------|----------|----------|----------------|-----------|
| O1  | 0.65970  | 0.27504  | 0.27887  | 0.0435 (4)     | 0.428 (2) |
| O2  | 0.73863  | 0.46240  | 0.24707  | 0.0406 (3)     |           |
| H2  | 0.7158   | 0.422    | 0.2746   | 0.061*         |           |
| O3  | 0.73548  | 0.49314  | −0.02964 | 0.0318 (3)     |           |
| H3  | 0.7141   | 0.459    | −0.0788  | 0.048*         |           |
| O4  | 0.65513  | 0.32200  | −0.14377 | 0.0333 (3)     |           |
| N1  | 0.63242  | 0.2052   | 0.43483  | 0.0232 (2)     | 0.428 (2) |
| H1A | 0.6785   | 0.167    | 0.462    | 0.028*         | 0.428 (2) |
| H1B | 0.636    | 0.252    | 0.3924   | 0.028*         | 0.428 (2) |
| N1B | 0.6296   | 0.2240   | 0.6962   | 0.0255 (3)     | 0.072 (2) |
| H1C | 0.676    | 0.184    | 0.716    | 0.031*         | 0.072 (2) |
| H1D | 0.625    | 0.261    | 0.741    | 0.031*         | 0.072 (2) |
| C1  | 0.63682  | 0.23098  | 0.20409  | 0.0303 (4)     |           |
| C2  | 0.58258  | 0.1108   | 0.17814  | 0.0392 (4)     |           |
| H2A | 0.53596  | 0.134104 | 0.126697 | 0.059*         |           |
| H2B | 0.567315 | 0.086926 | 0.225370 | 0.059*         |           |
| H2C | 0.609006 | 0.032779 | 0.165398 | 0.059*         |           |
| C3  | 0.66261  | 0.29590  | 0.14205  | 0.0232 (3)     |           |
| C4  | 0.71264  | 0.41203  | 0.16646  | 0.0264 (3)     |           |
| C5  | 0.73613  | 0.47613  | 0.10833  | 0.0265 (3)     |           |
| H5  | 0.769710 | 0.553232 | 0.125827 | 0.032*         |           |
| C6  | 0.71078  | 0.42813  | 0.20247  | 0.0228 (3)     |           |
| C7  | 0.66054  | 0.31225  | −0.00255 | 0.0194 (3)     |           |
| C8  | 0.63794  | 0.24981  | 0.05741  | 0.0206 (3)     |           |
| H8  | 0.604356 | 0.172691 | 0.039992 | 0.025*         |           |
| C9  | 0.63396  | 0.26350  | −0.09192 | 0.0242 (3)     |           |
| C10 | 0.58093  | 0.1419   | −0.12108 | 0.0339 (4)     |           |
| H10A| 0.568838 | 0.123625 | −0.182006| 0.051*         |           |
| H10B| 0.532509 | 0.160674 | −0.113824| 0.051*         |           |
| H10C| 0.606809 | 0.062098 | −0.086448| 0.051*         |           |
| C11 | 0.60624  | 0.27031  | 0.48402  | 0.0237 (3)     |           |
| H11 | 0.624879 | 0.227360 | 0.445699 | 0.028*         | 0.572 (2) |
### Atomic displacement parameters (Å²)

| \( U^{11} \) | \( U^{22} \) | \( U^{33} \) | \( U^{12} \) | \( U^{13} \) | \( U^{23} \) |
|---|---|---|---|---|---|
| O1 0.0494 (8) | 0.0610 (10) | 0.0232 (6) | 0.0200 (7) | 0.0178 (6) | 0.0096 (6) |
| O2 0.0380 (7) | 0.0502 (9) | 0.0258 (6) | 0.0040 (6) | 0.0048 (5) | −0.0143 (6) |
| O3 0.0326 (6) | 0.0283 (6) | 0.0415 (7) | −0.0002 (5) | 0.0223 (6) | 0.0058 (5) |
| O4 0.0363 (6) | 0.0425 (7) | 0.0241 (6) | 0.0130 (6) | 0.0153 (5) | 0.0058 (5) |
| N1 0.0294 (15) | 0.0217 (14) | 0.0224 (14) | 0.0077 (11) | 0.0147 (12) | 0.0002 (11) |
| N1B 0.0237 (7) | 0.0230 (7) | 0.0254 (7) | −0.0008 (5) | 0.0052 (6) | 0.0077 (6) |
| C1 0.0299 (8) | 0.0382 (9) | 0.0254 (7) | 0.0158 (7) | 0.0137 (6) | 0.0107 (7) |
| C2 0.0421 (10) | 0.0399 (10) | 0.0448 (10) | 0.0109 (8) | 0.0270 (9) | 0.0204 (8) |
| C3 0.0221 (6) | 0.0265 (7) | 0.0211 (6) | 0.0074 (5) | 0.0087 (5) | 0.0025 (5) |
| C4 0.0207 (7) | 0.0296 (8) | 0.0242 (7) | 0.0067 (6) | 0.0039 (5) | −0.0056 (6) |
| C5 0.0188 (6) | 0.0233 (7) | 0.0346 (8) | −0.0001 (5) | 0.0078 (6) | −0.0063 (6) |
| C6 0.0176 (6) | 0.0205 (6) | 0.0314 (7) | 0.0043 (5) | 0.0111 (5) | 0.0021 (6) |
| C7 0.0170 (6) | 0.0205 (6) | 0.0202 (6) | 0.0038 (5) | 0.0071 (5) | −0.0006 (5) |
| C8 0.0189 (6) | 0.0194 (6) | 0.0236 (7) | 0.0027 (5) | 0.0086 (5) | 0.0001 (5) |
| C9 0.0211 (6) | 0.0274 (7) | 0.0221 (7) | 0.0090 (5) | 0.0067 (5) | −0.0008 (6) |
| C10 0.0278 (8) | 0.0369 (9) | 0.0321 (8) | 0.0013 (7) | 0.0067 (6) | −0.0151 (7) |
| C11 0.0222 (6) | 0.0194 (6) | 0.0305 (7) | −0.0009 (5) | 0.0117 (6) | −0.0020 (6) |
| C12 0.0236 (7) | 0.0205 (7) | 0.0372 (9) | 0.0021 (5) | 0.0084 (6) | 0.0054 (6) |
| C13 0.0237 (7) | 0.0230 (7) | 0.0254 (7) | −0.0008 (5) | 0.0052 (6) | 0.0077 (6) |
| C14 0.0184 (6) | 0.0217 (7) | 0.0205 (7) | −0.0035 (5) | 0.0065 (5) | 0.0032 (5) |
| C15 0.0240 (7) | 0.0291 (8) | 0.0178 (6) | −0.0041 (6) | 0.0074 (5) | 0.0021 (5) |
| C16 0.0241 (7) | 0.0275 (7) | 0.0190 (6) | −0.0045 (6) | 0.0100 (5) | −0.0016 (5) |
| C17 0.0188 (6) | 0.0187 (6) | 0.0213 (6) | −0.0036 (5) | 0.0081 (5) | −0.0010 (5) |
| C18 0.0157 (6) | 0.0171 (6) | 0.0186 (6) | −0.0036 (5) | 0.0060 (5) | −0.0002 (5) |

### Geometric parameters (Å, °)

|       |       |       |       |
|-------|-------|-------|-------|
| O1—C1 | 1.240 (2) | C7—C8 | 1.389 (2) |
| O2—C4 | 1.3450 (19) | C7—C9 | 1.470 (2) |
| O3—H2 | 0.84 (3) | C8—H8 | 0.9500 |
| O3—C6 | 1.3418 (19) | C9—C10 | 1.500 (2) |
| O3—H3 | 0.83 (3) | C10—H10A | 0.9800 |
| O4—C9 | 1.240 (2) | C10—H10B | 0.9800 |

Acta Cryst. (2022). E78, 679-681
N1—C11 1.292 (3) C10—H10C 0.9800
N1—H1A 0.882 (10) C11—C12 1.396 (2)
N1—H1B 0.875 (10) C11—C17i 1.414 (2)
N1B—C13 1.243 (14) C11—H11 0.9500
N1B—H1C 0.882 (10) C12—C13 1.379 (2)
N1B—H1D 0.880 (10) C12—H12 0.9500
C1—C3 1.467 (2) C13—C14 1.408 (2)
C1—C2 1.500 (3) C13—H13 0.9500
C2—H2A 0.9800 C14—C18i 1.4210 (19)
C2—H2B 0.9800 C14—C15 1.432 (2)
C2—H2C 0.9800 C15—C16 1.360 (2)
C3—C8 1.392 (2) C15—H15 0.9500
C3—C4 1.424 (2) C16—C17 1.434 (2)
C4—C5 1.380 (2) C16—H16 0.9500
C5—C6 1.386 (2) C17—C18 1.4220 (19)
C5—H5 0.9500 C18—C18i 1.431 (3)
C6—C7 1.426 (2)

C4—O2—H2 109.5 O4—C9—C10 119.42 (15)
C6—O3—H3 109.5 C7—C9—C10 120.08 (14)
C11—N1—H1A 114 (2) C9—C10—H10A 109.5
C11—N1—H1B 116 (2) C9—C10—H10B 109.5
H1A—N1—H1B 106 (4) C9—C10—H10C 109.5
C13—N1B—H1C 120 (3) C10—C9—C10A 109.5
C13—N1B—H1D 120 (3) H10A—C10—H10B 109.5
H1C—N1B—H1D 106 (5) H10A—C10—H10C 109.5
O1—C1—C3 120.23 (18) N1—C11—C12 116.75 (19)
O1—C1—C2 119.14 (16) N1—C11—C17i 123.18 (19)
C3—C1—C2 120.63 (15) C12—C11—C17i 120.05 (14)
C1—C2—H2A 109.5 C12—C11—H11 120.0
C1—C2—H2B 109.5 C17i—C11—H11 120.0
H2A—C2—H2B 109.5 C13—C12—C11 121.28 (14)
C1—C2—H2C 109.5 C13—C12—H12 119.4
H2A—C2—H2C 109.5 C11—C12—H12 119.4
H2B—C2—H2C 109.5 N1B—C13—C14 111.3 (10)
C8—C3—C4 117.66 (14) N1B—C13—C14 127.6 (10)
C8—C3—C1 121.95 (15) C12—C13—C14 120.49 (14)
C4—C3—C1 120.37 (15) C12—C13—H13 119.8
O2—C4—C5 117.95 (16) C14—C13—H13 119.8
O2—C4—C3 120.93 (16) C13—C14—C18i 119.12 (14)
C5—C4—C3 121.12 (14) C13—C14—C15 122.02 (14)
C4—C5—C6 120.08 (15) C18i—C14—C15 118.86 (13)
C4—C5—H5 120.0 C16—C15—C14 121.37 (14)
C6—C5—H5 120.0 C16—C15—H15 119.3
O3—C6—C5 118.00 (15) C14—C15—H15 119.3
O3—C6—C7 121.48 (14) C15—C16—C17 121.20 (14)
C5—C6—C7 120.52 (14) C15—C16—H16 119.4
C8—C7—C6 118.10 (13) C17—C16—H16 119.4
C8—C7—C9          122.26 (14)        C11—C17—C18          118.88 (13)
C6—C7—C9          119.65 (14)        C11—C17—C16          122.40 (14)
C7—C8—C3          122.53 (14)        C18—C17—C16          118.72 (13)
C7—C8—H8          118.7            C14—C18—C17          120.17 (13)
C3—C8—H8          118.7            C14—C18—C18          119.80 (16)
O4—C9—C7          120.50 (15)        C17—C18—C18          120.04 (16)

Symmetry code: (i) −x+1, −y+1, −z+1.

Hydrogen-bond geometry (Å, °)

| D—H—A           | D—H  | H···A  | D···A  | D—H···A  |
|-----------------|-------|--------|--------|----------|
| O2—H2—O1       | 0.85  | 1.80   | 2.545  | 146      |
| O3—H3—O4       | 0.84  | 1.80   | 2.542  | 147      |
| N1—H1A···O3ii   | 0.88  | 2.30   | 3.131  | 156      |
| N1—H1B···O1     | 0.88  | 2.16   | 2.966  | 154      |
| N1B—H1C···O2ii  | 0.88  | 2.05   | 2.902  | 152      |
| N1B—H1D···O4iv  | 0.88  | 1.89   | 2.731  | 159      |

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