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Dimethyl 1-(3-cyanobenzyl)-1H-pyrazole-3,5-dicarboxylate

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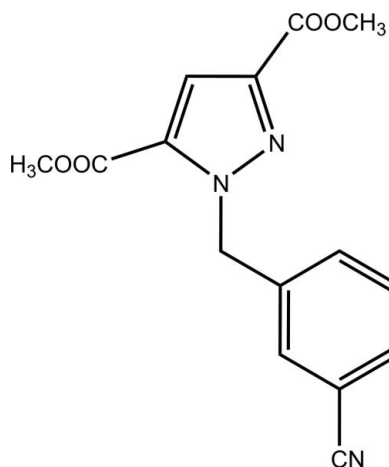
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Key indicators: single-crystal X-ray study; $T = 292$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.056; wR factor = 0.147; data-to-parameter ratio = 16.6.

In the molecule of the title compound, $\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_4$, the dihedral angle between the pyrazole and benzene ring planes is $67.7(1)^\circ$. The crystal structure is stabilized by an intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond and two weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

For the biological activity of pyrazoles, see: Lee *et al.* (1989); Chambers *et al.* (1985). For the importance of nitrile derivatives in the synthesis of some heterocyclic molecules, see: Radl *et al.* (2000). For related structures, see: Fu & Zhao (2007); Xiao & Zhao (2008a,b,c).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_4$
 $M_r = 299.28$
Triclinic, $P\bar{1}$
 $a = 8.783(3)$ Å
 $b = 9.538(4)$ Å
 $c = 9.999(4)$ Å
 $\alpha = 68.42(3)^\circ$
 $\beta = 71.79(4)^\circ$
 $\gamma = 82.13(4)^\circ$
 $V = 739.7(5)$ Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 292$ K
 $0.40 \times 0.30 \times 0.20$ mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.968$, $T_{\max} = 0.979$
7555 measured reflections
3338 independent reflections
2093 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.147$
 $S = 1.05$
3338 reflections
201 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.16$ e Å⁻³
 $\Delta\rho_{\min} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{C15}-\text{H15B}\cdots\text{O1}^{\text{i}}$ | 0.96 | 2.43 | 3.363 (3) | 163 |
| $\text{C9}-\text{H9}\cdots\text{O1}^{\text{ii}}$ | 0.93 | 2.53 | 3.348 (3) | 147 |
| $\text{C6}-\text{H6A}\cdots\text{O4}$ | 0.97 | 2.41 | 2.979 (3) | 117 |

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2204).

References

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supporting information

Acta Cryst. (2009). E65, o1132 [doi:10.1107/S1600536809014895]

Dimethyl 1-(3-cyanobenzyl)-1*H*-pyrazole-3,5-dicarboxylate

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S1. Comment

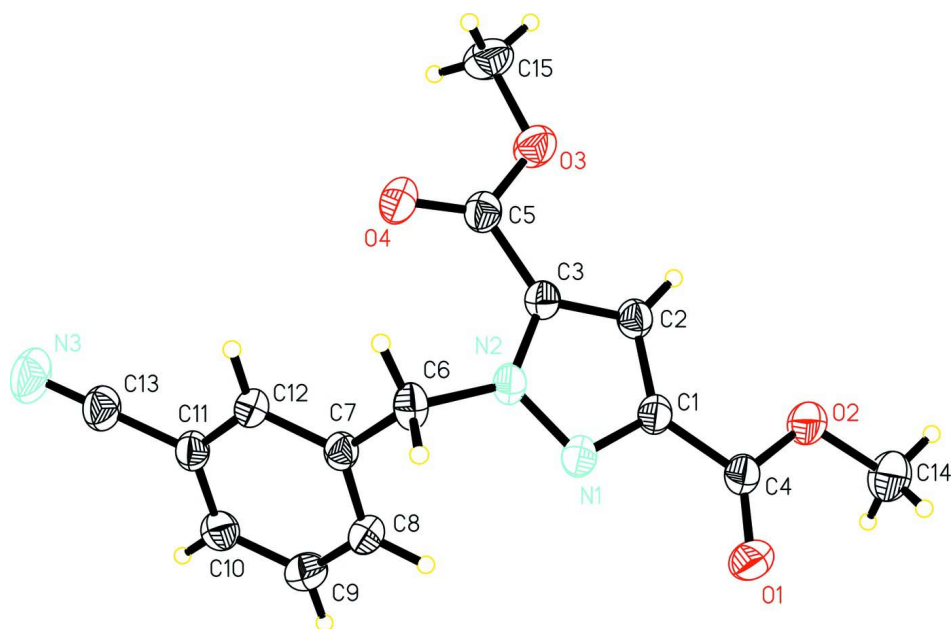
It is well known that many pyrazole-related molecules have received much attention due to their biological activities (Lee *et al.*, 1989; Chambers *et al.*, 1985). In addition, nitrile derivatives are important materials in the synthesis of some heterocyclic molecules (Radl *et al.*, 2000). Recently, we have reported a few benzonitrile compounds (Fu *et al.*, 2007; Xiao *et al.*, 2008a, 2008b, 2008c). As an extension of our work on the structural characterization of nitrile compounds, the structure of the title compound is reported here. In the molecule of the title compound (Fig. 1) bond lengths and angles have normal values. The dihedral angle between the planes of the pyrazole and phenyl rings is 67.74 (14) °. The molecular conformation is stabilized by an intramolecular C—H···O hydrogen bond and weak intermolecular C—H···O interactions (Table 1).

S2. Experimental

Dimethyl 1*H*-Pyrazole-3,5-dicarboxylate (0.185 mg, 1 mmol) and 3-(bromomethyl)benzonitrile (0.196 mg, 1 mmol) were dissolved in acetone in the presence of K₂CO₃ (0.138 mg, 1 mmol) and heated under reflux for 1 d. After the mixture was cooled to room temperature, the solution was filtered and the solvent removed in vacuum to afford a white precipitate of the title compound. Colourless crystals suitable for X-ray diffraction were obtained from a solution of 100 mg in 15 ml diethylether by slow evaporation after 7 d.

S3. Refinement

All H atoms were detected in a difference Fourier map but were placed in calculated positions and refined using a riding motion approximation, with C_{aryl}—H, C_{methylene}—H, C_{methyl}—H = 0.93, 0.97 and 0.96 Å. $U_{\text{iso}}(\text{H}_{\text{aryl}}) = 1.2U_{\text{eq}}(\text{C}_{\text{aryl}})$; $U_{\text{iso}}(\text{H}_{\text{methylene}}) = 1.2U_{\text{eq}}(\text{C}_{\text{methylene}})$; $U_{\text{iso}}(\text{H}_{\text{methyl}}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

**Figure 1**

The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Dimethyl 1-(3-cyanobenzyl)-1H-pyrazole-3,5-dicarboxylate

Crystal data

$C_{15}H_{13}N_3O_4$

$M_r = 299.28$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.783$ (3) Å

$b = 9.538$ (4) Å

$c = 9.999$ (4) Å

$\alpha = 68.42$ (3)°

$\beta = 71.79$ (4)°

$\gamma = 82.13$ (4)°

$V = 739.7$ (5) Å³

$Z = 2$

$F(000) = 312$

$D_x = 1.344$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1442 reflections

$\theta = 2.4$ – 27.3 °

$\mu = 0.10$ mm⁻¹

$T = 292$ K

Block, colorless

$0.40 \times 0.30 \times 0.20$ mm

Data collection

Rigaku SCXmini

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 13.6612 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2005)

$T_{\min} = 0.968$, $T_{\max} = 0.979$

7555 measured reflections

3338 independent reflections

2093 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 27.4$ °, $\theta_{\min} = 2.3$ °

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.147$
 $S = 1.05$
 3338 reflections
 201 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
 $w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.0696P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|-------------|------------|----------------------------------|
| C1 | 0.2466 (2) | 0.1608 (2) | 0.4437 (2) | 0.0473 (5) |
| C2 | 0.2324 (2) | 0.0151 (2) | 0.4487 (2) | 0.0485 (5) |
| H2 | 0.2544 | -0.0753 | 0.5185 | 0.058* |
| C3 | 0.1787 (2) | 0.0343 (2) | 0.3276 (2) | 0.0453 (5) |
| C4 | 0.2972 (2) | 0.2120 (3) | 0.5461 (2) | 0.0530 (5) |
| C5 | 0.1399 (2) | -0.0794 (2) | 0.2763 (2) | 0.0516 (5) |
| C6 | 0.1126 (2) | 0.2702 (2) | 0.1210 (2) | 0.0513 (5) |
| H6A | 0.1388 | 0.2105 | 0.0561 | 0.062* |
| H6B | 0.1713 | 0.3630 | 0.0669 | 0.062* |
| C7 | -0.0655 (2) | 0.3076 (2) | 0.1568 (2) | 0.0441 (5) |
| C8 | -0.1362 (3) | 0.3905 (2) | 0.2511 (2) | 0.0548 (5) |
| H8 | -0.0735 | 0.4208 | 0.2948 | 0.066* |
| C9 | -0.2976 (3) | 0.4282 (3) | 0.2806 (3) | 0.0618 (6) |
| H9 | -0.3429 | 0.4818 | 0.3453 | 0.074* |
| C10 | -0.3928 (3) | 0.3868 (2) | 0.2143 (2) | 0.0564 (6) |
| H10 | -0.5012 | 0.4135 | 0.2327 | 0.068* |
| C11 | -0.3234 (2) | 0.3050 (2) | 0.1206 (2) | 0.0501 (5) |
| C12 | -0.1607 (2) | 0.2639 (2) | 0.0923 (2) | 0.0476 (5) |
| H12 | -0.1163 | 0.2074 | 0.0303 | 0.057* |
| C13 | -0.4218 (3) | 0.2621 (3) | 0.0498 (3) | 0.0625 (6) |
| C14 | 0.3840 (4) | 0.1361 (3) | 0.7627 (3) | 0.0947 (10) |
| H14A | 0.4685 | 0.2077 | 0.7128 | 0.142* |
| H14B | 0.4199 | 0.0470 | 0.8309 | 0.142* |
| H14C | 0.2922 | 0.1797 | 0.8174 | 0.142* |
| C15 | 0.1386 (3) | -0.3432 (3) | 0.3310 (3) | 0.0756 (7) |

| | | | | |
|------|--------------|---------------|--------------|------------|
| H15A | 0.0277 | -0.3388 | 0.3343 | 0.113* |
| H15B | 0.1612 | -0.4358 | 0.4042 | 0.113* |
| H15C | 0.2046 | -0.3388 | 0.2329 | 0.113* |
| N1 | 0.2055 (2) | 0.26560 (19) | 0.32636 (19) | 0.0517 (4) |
| N2 | 0.16398 (19) | 0.18644 (18) | 0.25641 (17) | 0.0460 (4) |
| N3 | -0.4990 (3) | 0.2293 (3) | -0.0067 (3) | 0.0878 (7) |
| O1 | 0.2979 (2) | 0.34090 (19) | 0.5354 (2) | 0.0816 (6) |
| O2 | 0.3407 (2) | 0.09684 (17) | 0.65206 (18) | 0.0724 (5) |
| O3 | 0.1723 (2) | -0.21721 (16) | 0.36374 (17) | 0.0659 (5) |
| O4 | 0.0863 (2) | -0.05489 (18) | 0.1728 (2) | 0.0769 (5) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0473 (12) | 0.0513 (12) | 0.0480 (12) | -0.0011 (9) | -0.0202 (9) | -0.0170 (9) |
| C2 | 0.0510 (12) | 0.0492 (12) | 0.0461 (12) | -0.0012 (9) | -0.0194 (9) | -0.0126 (9) |
| C3 | 0.0429 (11) | 0.0480 (11) | 0.0461 (11) | -0.0024 (8) | -0.0145 (9) | -0.0153 (9) |
| C4 | 0.0528 (13) | 0.0599 (14) | 0.0562 (13) | 0.0044 (10) | -0.0243 (10) | -0.0258 (11) |
| C5 | 0.0471 (12) | 0.0576 (13) | 0.0532 (13) | -0.0022 (9) | -0.0164 (10) | -0.0206 (10) |
| C6 | 0.0494 (12) | 0.0601 (13) | 0.0435 (12) | -0.0053 (9) | -0.0207 (9) | -0.0094 (9) |
| C7 | 0.0505 (12) | 0.0415 (11) | 0.0400 (11) | -0.0054 (8) | -0.0182 (9) | -0.0078 (8) |
| C8 | 0.0627 (14) | 0.0541 (13) | 0.0594 (13) | -0.0035 (10) | -0.0279 (11) | -0.0232 (10) |
| C9 | 0.0677 (15) | 0.0597 (14) | 0.0658 (15) | 0.0024 (11) | -0.0194 (12) | -0.0314 (12) |
| C10 | 0.0512 (13) | 0.0570 (13) | 0.0634 (14) | 0.0023 (10) | -0.0194 (11) | -0.0219 (11) |
| C11 | 0.0499 (12) | 0.0522 (12) | 0.0526 (12) | -0.0029 (9) | -0.0233 (10) | -0.0151 (9) |
| C12 | 0.0529 (12) | 0.0511 (12) | 0.0459 (11) | -0.0005 (9) | -0.0206 (9) | -0.0195 (9) |
| C13 | 0.0509 (13) | 0.0758 (16) | 0.0698 (16) | 0.0021 (11) | -0.0254 (12) | -0.0295 (12) |
| C14 | 0.140 (3) | 0.098 (2) | 0.0809 (19) | 0.0188 (18) | -0.075 (2) | -0.0427 (16) |
| C15 | 0.097 (2) | 0.0552 (15) | 0.0804 (18) | -0.0088 (13) | -0.0245 (15) | -0.0280 (13) |
| N1 | 0.0544 (10) | 0.0532 (10) | 0.0552 (11) | -0.0020 (8) | -0.0259 (8) | -0.0185 (8) |
| N2 | 0.0464 (9) | 0.0503 (10) | 0.0446 (9) | -0.0034 (7) | -0.0205 (7) | -0.0131 (7) |
| N3 | 0.0656 (14) | 0.124 (2) | 0.1012 (18) | 0.0028 (13) | -0.0437 (13) | -0.0557 (16) |
| O1 | 0.1237 (16) | 0.0589 (11) | 0.0919 (13) | 0.0097 (10) | -0.0644 (12) | -0.0358 (9) |
| O2 | 0.1047 (14) | 0.0650 (10) | 0.0687 (11) | 0.0110 (9) | -0.0542 (10) | -0.0268 (8) |
| O3 | 0.0894 (12) | 0.0511 (9) | 0.0636 (10) | -0.0076 (8) | -0.0321 (9) | -0.0164 (7) |
| O4 | 0.1025 (14) | 0.0705 (11) | 0.0837 (12) | 0.0033 (9) | -0.0579 (11) | -0.0317 (9) |

Geometric parameters (Å, °)

| | | | |
|-------|-----------|---------|-----------|
| C1—N1 | 1.345 (2) | C8—H8 | 0.9300 |
| C1—C2 | 1.393 (3) | C9—C10 | 1.387 (3) |
| C1—C4 | 1.482 (3) | C9—H9 | 0.9300 |
| C2—C3 | 1.375 (2) | C10—C11 | 1.381 (3) |
| C2—H2 | 0.9300 | C10—H10 | 0.9300 |
| C3—N2 | 1.369 (2) | C11—C12 | 1.397 (3) |
| C3—C5 | 1.475 (3) | C11—C13 | 1.452 (3) |
| C4—O1 | 1.195 (2) | C12—H12 | 0.9300 |
| C4—O2 | 1.325 (2) | C13—N3 | 1.143 (3) |

| | | | |
|-------------|--------------|----------------|--------------|
| C5—O4 | 1.201 (2) | C14—O2 | 1.455 (3) |
| C5—O3 | 1.334 (3) | C14—H14A | 0.9600 |
| C6—N2 | 1.471 (2) | C14—H14B | 0.9600 |
| C6—C7 | 1.514 (3) | C14—H14C | 0.9600 |
| C6—H6A | 0.9700 | C15—O3 | 1.444 (3) |
| C6—H6B | 0.9700 | C15—H15A | 0.9600 |
| C7—C12 | 1.385 (2) | C15—H15B | 0.9600 |
| C7—C8 | 1.395 (3) | C15—H15C | 0.9600 |
| C8—C9 | 1.380 (3) | N1—N2 | 1.347 (2) |
| N1—C1—C2 | 111.68 (17) | C10—C9—H9 | 119.8 |
| N1—C1—C4 | 118.44 (18) | C11—C10—C9 | 118.7 (2) |
| C2—C1—C4 | 129.88 (18) | C11—C10—H10 | 120.6 |
| C3—C2—C1 | 104.90 (17) | C9—C10—H10 | 120.6 |
| C3—C2—H2 | 127.6 | C10—C11—C12 | 121.23 (18) |
| C1—C2—H2 | 127.6 | C10—C11—C13 | 119.1 (2) |
| N2—C3—C2 | 107.05 (17) | C12—C11—C13 | 119.63 (19) |
| N2—C3—C5 | 123.21 (17) | C7—C12—C11 | 119.83 (19) |
| C2—C3—C5 | 129.74 (18) | C7—C12—H12 | 120.1 |
| O1—C4—O2 | 124.09 (19) | C11—C12—H12 | 120.1 |
| O1—C4—C1 | 124.3 (2) | N3—C13—C11 | 179.5 (3) |
| O2—C4—C1 | 111.57 (19) | O2—C14—H14A | 109.5 |
| O4—C5—O3 | 124.0 (2) | O2—C14—H14B | 109.5 |
| O4—C5—C3 | 126.4 (2) | H14A—C14—H14B | 109.5 |
| O3—C5—C3 | 109.59 (18) | O2—C14—H14C | 109.5 |
| N2—C6—C7 | 112.47 (16) | H14A—C14—H14C | 109.5 |
| N2—C6—H6A | 109.1 | H14B—C14—H14C | 109.5 |
| C7—C6—H6A | 109.1 | O3—C15—H15A | 109.5 |
| N2—C6—H6B | 109.1 | O3—C15—H15B | 109.5 |
| C7—C6—H6B | 109.1 | H15A—C15—H15B | 109.5 |
| H6A—C6—H6B | 107.8 | O3—C15—H15C | 109.5 |
| C12—C7—C8 | 118.64 (19) | H15A—C15—H15C | 109.5 |
| C12—C7—C6 | 120.28 (18) | H15B—C15—H15C | 109.5 |
| C8—C7—C6 | 121.05 (17) | C1—N1—N2 | 104.90 (16) |
| C9—C8—C7 | 121.12 (19) | N1—N2—C3 | 111.47 (15) |
| C9—C8—H8 | 119.4 | N1—N2—C6 | 118.28 (16) |
| C7—C8—H8 | 119.4 | C3—N2—C6 | 130.24 (16) |
| C8—C9—C10 | 120.4 (2) | C4—O2—C14 | 115.33 (19) |
| C8—C9—H9 | 119.8 | C5—O3—C15 | 117.10 (18) |
| N1—C1—C2—C3 | -0.4 (2) | C9—C10—C11—C13 | -179.5 (2) |
| C4—C1—C2—C3 | 179.1 (2) | C8—C7—C12—C11 | 0.9 (3) |
| C1—C2—C3—N2 | 0.3 (2) | C6—C7—C12—C11 | -177.04 (18) |
| C1—C2—C3—C5 | -179.23 (19) | C10—C11—C12—C7 | -1.1 (3) |
| N1—C1—C4—O1 | 3.2 (3) | C13—C11—C12—C7 | 178.51 (18) |
| C2—C1—C4—O1 | -176.3 (2) | C2—C1—N1—N2 | 0.3 (2) |
| N1—C1—C4—O2 | -177.21 (18) | C4—C1—N1—N2 | -179.24 (16) |
| C2—C1—C4—O2 | 3.3 (3) | C1—N1—N2—C3 | -0.1 (2) |

| | | | |
|----------------|--------------|--------------|--------------|
| N2—C3—C5—O4 | -3.1 (3) | C1—N1—N2—C6 | -179.84 (16) |
| C2—C3—C5—O4 | 176.4 (2) | C2—C3—N2—N1 | -0.1 (2) |
| N2—C3—C5—O3 | 177.39 (18) | C5—C3—N2—N1 | 179.45 (17) |
| C2—C3—C5—O3 | -3.1 (3) | C2—C3—N2—C6 | 179.55 (18) |
| N2—C6—C7—C12 | -124.57 (19) | C5—C3—N2—C6 | -0.9 (3) |
| N2—C6—C7—C8 | 57.5 (2) | C7—C6—N2—N1 | -87.7 (2) |
| C12—C7—C8—C9 | 0.2 (3) | C7—C6—N2—C3 | 92.6 (2) |
| C6—C7—C8—C9 | 178.21 (19) | O1—C4—O2—C14 | 2.8 (4) |
| C7—C8—C9—C10 | -1.3 (3) | C1—C4—O2—C14 | -176.8 (2) |
| C8—C9—C10—C11 | 1.1 (3) | O4—C5—O3—C15 | -0.6 (3) |
| C9—C10—C11—C12 | 0.1 (3) | C3—C5—O3—C15 | 178.93 (18) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| C15—H15B...O1 ⁱ | 0.96 | 2.43 | 3.363 (3) | 163 |
| C9—H9...O1 ⁱⁱ | 0.93 | 2.53 | 3.348 (3) | 147 |
| C6—H6A...O4 | 0.97 | 2.41 | 2.979 (3) | 117 |

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