# organic compounds

# 1,3-Diallyl-6-bromo-1*H*-imidazo[4,5-*b*]-pyridin-2(3*H*)-one

**Siham Dahmani,**[a]*** Youssef Kandri Rodi,**[a] **Santiago V. Luis,**[b] **Michael Bolte**[c] **and Lahcen El Ammari**[d]

[a]Laboratoire de Chimie Organique Appliquée, Université Sidi Mohamed, Ben Abdallah, Faculté des Sciences et Techniques, Route d'Immouzzer, BP 2202 Fès, Morocco, [b]Departamento de Quimica Inorganica & Organica, ESTCE, Universitat Jaume I, E-12080 Castellon, Spain, [c]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany, and [d]Laboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Université Mohammed V-Agdal, Avenue Ibn Battouta, BP 1014, Rabat, Morocco
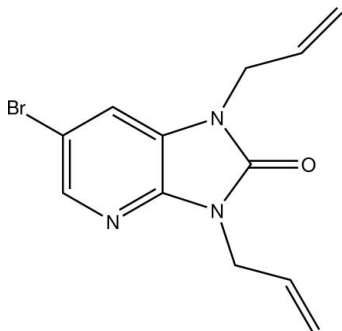Correspondence e-mail: s_dahmani12@yahoo.fr

In the molecule of the title compound, $C_{12}H_{12}BrN_3O$, the fused-ring system is essentially planar, the largest deviation from the mean plane being 0.0148 (3) Å. The two allyl groups are nearly perpendicular to the imidazo[4,5-*b*]pyridine plane [C—C—N—C torsion angles of 81.6 (4) and −77.2 (4)°] and point in the same direction. The planes through the atoms forming each allyl group are nearly perpendicular to the imidazo[4,5-*b*]pyridin-2-one system, as indicated by the dihedral angles between them of 80.8 (5) and 73.6 (5)°.

## Related literature

For background to the biological activity of substituted imidazopyridines and related compounds, see: Barraclough *et al.* (1990); Bavetsias *et al.* (2007, 2010); Coates *et al.* (1993); Liu *et al.* (2008); Ryabukhin *et al.* (2006); Schiffmann *et al.* (2006).



## Experimental

### Crystal data

| | |
|---|---|
| $C_{12}H_{12}BrN_3O$ | $V = 1281.56$ (11) Å³ |
| $M_r = 294.16$ | $Z = 4$ |
| Orthorhombic, $Pna2_1$ | Mo $K\alpha$ radiation |
| $a = 5.4110$ (3) Å | $\mu = 3.20$ mm⁻¹ |
| $b = 25.4205$ (12) Å | $T = 273$ K |
| $c = 9.3170$ (4) Å | $0.52 \times 0.32 \times 0.14$ mm |

### Data collection

| | |
|---|---|
| Bruker CCD three-circle diffractometer | 8789 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 1997) | 3201 independent reflections |
| | 2361 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.202$, $T_{max} = 0.800$ | $R_{int} = 0.027$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | H-atom parameters constrained |
| $wR(F^2) = 0.100$ | $\Delta\rho_{max} = 0.44$ e Å⁻³ |
| $S = 1.03$ | $\Delta\rho_{min} = -0.23$ e Å⁻³ |
| 3201 reflections | Absolute structure: Flack (1983), 1494 Friedel pairs |
| 156 parameters | Flack parameter: 0.040 (17) |
| 1 restraint | |

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2445).

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# supporting information

## 1,3-Diallyl-6-bromo-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

### Siham Dahmani, Youssef Kandri Rodi, Santiago V. Luis, Michael Bolte and Lahcen El Ammari

### S1. Comment

Substituted imidazopyridines and structurally related compounds are of pharmacological and therapeutical interest. They have been tested for their potential as anticancer, inotropic (Barraclough *et al.*, 1990), selective antihistamine (H1) agents and antibacterial activity (Liu *et al.*, 2008). Imidazo[4,5-*b*]pyridine derivatives were also reported as Aurora kinases (Bavetsias *et al.*, 2007); Bavetsias *et al.*, 2010), and cyclic PDE inhibitors (Coates *et al.*, 1993). The preparation of these compounds is usually straightforward, and a number of synthetic methods are already available (Ryabukhin *et al.*, 2006; Schiffmann *et al.*, 2006).

In this work, we report the synthesis of 1,3-diallyl-6-bromo-1,3-dihydroimidazo [4,5-*b*]pyridin-2-one *via* the reaction between 6-bromo-1,3-dihydro-imidazo [4,5 - b-]pyridin-2-one and allylbromide in DMF using $K_2CO_3$ as base (scheme1).
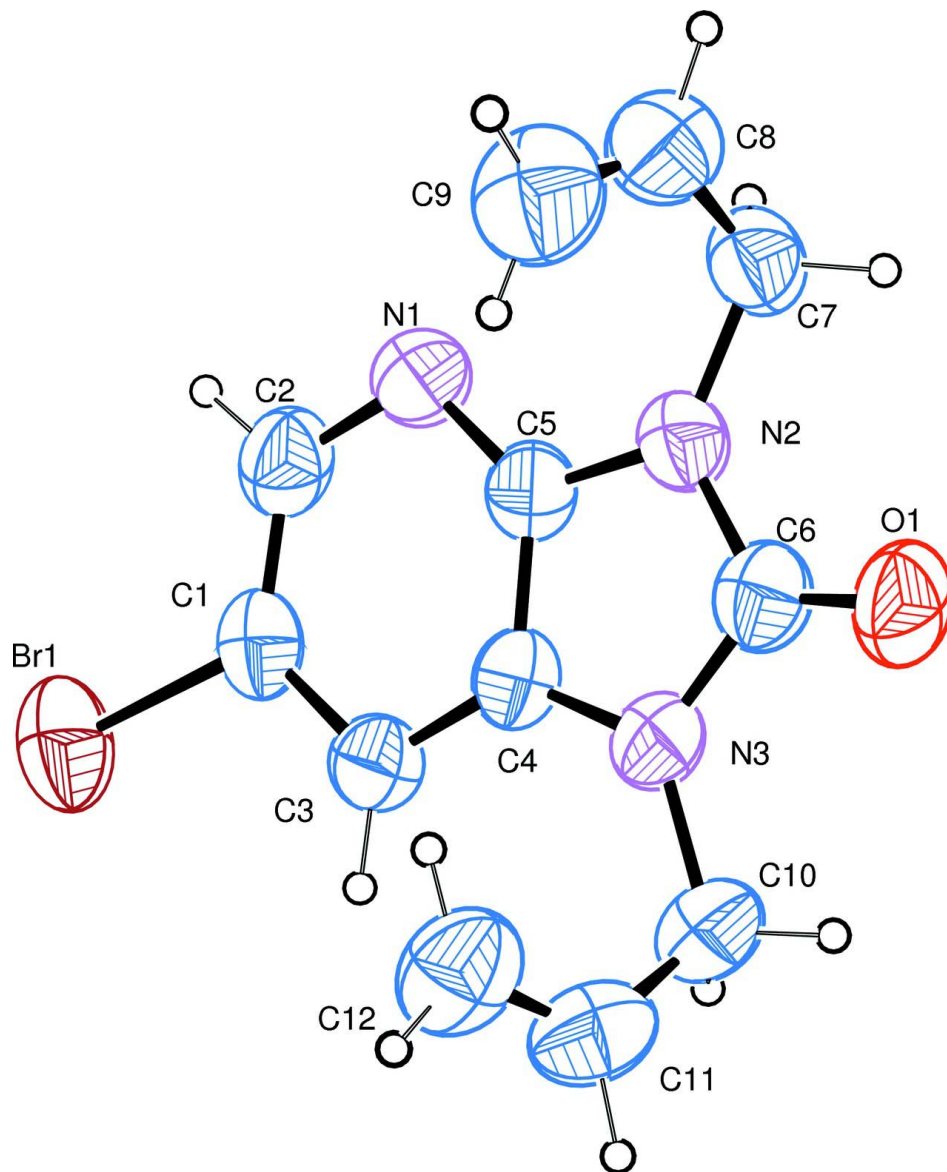
The Plot of the title compound molecule is shown in Fig.1. The two fused five and six-membered rings are nearly planar with the maximum deviation of -0.014 (3)Å from N1. The two allyl chains (–C7–C8–C9) and (–C10–C11–C12) are almost perpendicular to the imidazo[4,5-*b*]pyridine system mean plane as indicated by the following torsion angles C8–C7–N2–C5 and C11–C10–N3–C4 of 81.6 (4)° and -77.2 (4) ° respectively.

### S2. Experimental

To a stirred solution of 6-bromo-1,3-dihydro-imidazo[4,5 - b-]pyridin-2- one (0.5 g; 2.33 mmol), $K_2CO_3$ (1.29 g; 9.34 mmol), and tetrabutylammonium bromide (0.07 g; 2.37 10 $^{-4}$ mol) in DMF, allylbromide (0.5 ml; 5.84 mmol) was added dropwise. Stirring was continued at room temperature for 24 h. After completion of reaction (monitored by TLC), the salt was filtered and the solvent was removed under reduced pressure. The resulting residue was purified by column chromatography on silica gel using ethylacetate/hexane (1/1) as eluent. The crystals of the title compound are obtained by dissolving 80 mg of product in 4 ml of ethanol at about 353 K, followed by a slow evaporation of the solvent.

### S3. Refinement

The origin of the non centro symmetric space group is fxed by the *SHELXL* program and the 1495 Friedel opposite reflections are not merged. H atoms were located in a difference map and treated as riding with C—H = 0.93 Å and 0.97 Å for aromatic and methylene respectively. All H atoms with $U_{iso}(H) = 1.2 U_{eq}$ (aromatic and methylene).

**Figure 1**

Plot of the molecule of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

**1,3-Diallyl-6-bromo-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one**

*Crystal data*

| | |
|---|---|
| $C_{12}H_{12}BrN_3O$ | $F(000) = 592$ |
| $M_r = 294.16$ | $D_x = 1.525$ Mg m$^{-3}$ |
| Orthorhombic, $Pna2_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: p 2c -2n | Cell parameters from 3201 reflections |
| $a = 5.4110$ (3) Å | $\theta = 1.6$–28.5° |
| $b = 25.4205$ (12) Å | $\mu = 3.20$ mm$^{-1}$ |
| $c = 9.3170$ (4) Å | $T = 273$ K |
| $V = 1281.56$ (11) Å$^3$ | Block, colourless |
| $Z = 4$ | $0.52 \times 0.32 \times 0.14$ mm |

*Data collection*

| | |
|---|---|
| Bruker CCD three-circle diffractometer | 8789 measured reflections |
| Radiation source: sealed tube | 3201 independent reflections |
| Graphite monochromator | 2361 reflections with $I > 2\sigma(I)$ |
| phi and $\omega$ scans | $R_{int} = 0.027$ |
| Absorption correction: multi-scan ($SADABS$; Bruker, 1997) | $\theta_{max} = 28.5°$, $\theta_{min} = 1.6°$ |
| | $h = -6\rightarrow7$ |
| $T_{min} = 0.202$, $T_{max} = 0.800$ | $k = -25\rightarrow34$ |
| | $l = -12\rightarrow12$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | H-atom parameters constrained |
| $wR(F^2) = 0.100$ | $w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.2176P]$ |
| $S = 1.03$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3201 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 156 parameters | $\Delta\rho_{max} = 0.44$ e Å$^{-3}$ |
| 1 restraint | $\Delta\rho_{min} = -0.23$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 1494 Friedel pairs |
| Secondary atom site location: difference Fourier map | Absolute structure parameter: 0.040 (17) |

*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 Rw factor 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 0.99838 (6) | 0.148410 (15) | 0.99886 (11) | 0.07396 (14) |
| N1 | 0.5607 (5) | 0.19539 (10) | 0.6617 (3) | 0.0585 (6) |
| N2 | 0.2257 (4) | 0.14754 (9) | 0.5520 (3) | 0.0541 (6) |
| N3 | 0.2480 (5) | 0.07428 (9) | 0.6813 (3) | 0.0552 (6) |
| O1 | −0.0599 (4) | 0.08103 (10) | 0.5080 (4) | 0.0730 (6) |
| C1 | 0.7552 (5) | 0.14820 (11) | 0.8523 (3) | 0.0515 (7) |
| C2 | 0.7283 (6) | 0.19139 (12) | 0.7678 (4) | 0.0592 (8) |
| H2 | 0.8321 | 0.2199 | 0.7842 | 0.071* |
| C3 | 0.6043 (6) | 0.10361 (12) | 0.8374 (3) | 0.0532 (7) |
| H3 | 0.6209 | 0.0739 | 0.8948 | 0.064* |
| C4 | 0.4298 (5) | 0.10753 (12) | 0.7306 (3) | 0.0496 (6) |
| C5 | 0.4169 (6) | 0.15329 (11) | 0.6493 (4) | 0.0489 (6) |
| C6 | 0.1188 (6) | 0.09843 (13) | 0.5717 (4) | 0.0579 (7) |
| C7 | 0.1361 (6) | 0.18683 (13) | 0.4530 (4) | 0.0621 (8) |
| H7A | 0.1349 | 0.2207 | 0.5011 | 0.075* |

| | | | |
|---|---|---|---|
| H7B | −0.0331 | 0.1784 | 0.4274 | 0.075* |
| C8 | 0.2846 (7) | 0.19138 (15) | 0.3195 (4) | 0.0706 (9) |
| H8 | 0.2371 | 0.2173 | 0.2548 | 0.090 (12)* |
| C9 | 0.4653 (9) | 0.1641 (3) | 0.2855 (6) | 0.0939 (15) |
| H9A | 0.5201 | 0.1376 | 0.3466 | 0.113* |
| H9B | 0.5461 | 0.1701 | 0.1989 | 0.113* |
| C10 | 0.1712 (7) | 0.02523 (13) | 0.7497 (4) | 0.0694 (9) |
| H10A | 0.0091 | 0.0156 | 0.7137 | 0.083* |
| H10B | 0.1562 | 0.0311 | 0.8522 | 0.083* |
| C11 | 0.3410 (8) | −0.01892 (15) | 0.7256 (5) | 0.0771 (10) |
| H11 | 0.3027 | −0.0503 | 0.7717 | 0.129 (19)* |
| C12 | 0.5362 (7) | −0.01882 (17) | 0.6479 (6) | 0.0832 (12) |
| H12A | 0.5828 | 0.0116 | 0.5995 | 0.100* |
| H12B | 0.6313 | −0.0491 | 0.6400 | 0.100* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.05433 (18) | 0.1026 (3) | 0.0649 (2) | 0.00262 (14) | −0.01189 (13) | −0.0127 (2) |
| N1 | 0.0564 (13) | 0.0460 (14) | 0.0731 (17) | −0.0070 (11) | −0.0083 (14) | 0.0032 (13) |
| N2 | 0.0478 (13) | 0.0545 (14) | 0.0599 (15) | −0.0015 (10) | −0.0090 (11) | 0.0029 (10) |
| N3 | 0.0493 (13) | 0.0476 (13) | 0.0686 (15) | −0.0089 (10) | −0.0042 (12) | 0.0025 (11) |
| O1 | 0.0564 (12) | 0.0847 (15) | 0.0781 (16) | −0.0178 (10) | −0.0148 (16) | −0.0040 (16) |
| C1 | 0.0412 (14) | 0.0642 (18) | 0.0489 (15) | 0.0007 (13) | −0.0024 (12) | −0.0096 (13) |
| C2 | 0.0507 (16) | 0.0556 (17) | 0.071 (2) | −0.0074 (13) | −0.0041 (15) | −0.0080 (15) |
| C3 | 0.0489 (15) | 0.0567 (17) | 0.0539 (16) | 0.0026 (13) | 0.0034 (14) | 0.0046 (13) |
| C4 | 0.0416 (14) | 0.0549 (17) | 0.0522 (16) | −0.0013 (11) | 0.0038 (12) | −0.0024 (13) |
| C5 | 0.0446 (14) | 0.0454 (16) | 0.0566 (17) | 0.0003 (11) | −0.0005 (14) | −0.0055 (13) |
| C6 | 0.0531 (17) | 0.0630 (18) | 0.0575 (17) | −0.0051 (14) | −0.0016 (15) | −0.0051 (15) |
| C7 | 0.0550 (17) | 0.0651 (19) | 0.0662 (19) | 0.0019 (13) | −0.0108 (15) | 0.0016 (15) |
| C8 | 0.077 (2) | 0.083 (2) | 0.0523 (17) | −0.0094 (19) | −0.0090 (18) | 0.0031 (17) |
| C9 | 0.092 (4) | 0.119 (4) | 0.071 (3) | 0.003 (2) | 0.017 (2) | −0.005 (3) |
| C10 | 0.065 (2) | 0.062 (2) | 0.081 (2) | −0.0164 (15) | 0.0003 (19) | 0.0097 (17) |
| C11 | 0.089 (3) | 0.056 (2) | 0.086 (3) | −0.0155 (18) | 0.004 (2) | 0.0058 (18) |
| C12 | 0.086 (3) | 0.066 (3) | 0.098 (3) | −0.0118 (17) | 0.008 (2) | −0.014 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C1 | 1.896 (3) | C4—C5 | 1.390 (4) |
| N1—C5 | 1.328 (4) | C7—C8 | 1.485 (5) |
| N1—C2 | 1.345 (4) | C7—H7A | 0.9700 |
| N2—C5 | 1.383 (4) | C7—H7B | 0.9700 |
| N2—C6 | 1.388 (4) | C8—C9 | 1.240 (6) |
| N2—C7 | 1.444 (4) | C8—H8 | 0.9300 |
| N3—C4 | 1.376 (4) | C9—H9A | 0.9300 |
| N3—C6 | 1.381 (4) | C9—H9B | 0.9300 |
| N3—C10 | 1.461 (4) | C10—C11 | 1.467 (6) |
| O1—C6 | 1.218 (4) | C10—H10A | 0.9700 |

| | | | |
|---|---|---|---|
| C1—C2 | 1.359 (4) | C10—H10B | 0.9700 |
| C1—C3 | 1.404 (4) | C11—C12 | 1.281 (6) |
| C2—H2 | 0.9300 | C11—H11 | 0.9300 |
| C3—C4 | 1.375 (5) | C12—H12A | 0.9300 |
| C3—H3 | 0.9300 | C12—H12B | 0.9300 |
| | | | |
| C5—N1—C2 | 113.5 (3) | N2—C7—C8 | 114.0 (3) |
| C5—N2—C6 | 108.7 (2) | N2—C7—H7A | 108.7 |
| C5—N2—C7 | 126.6 (2) | C8—C7—H7A | 108.7 |
| C6—N2—C7 | 124.5 (3) | N2—C7—H7B | 108.7 |
| C4—N3—C6 | 109.6 (3) | C8—C7—H7B | 108.7 |
| C4—N3—C10 | 125.6 (3) | H7A—C7—H7B | 107.6 |
| C6—N3—C10 | 123.9 (3) | C9—C8—C7 | 126.6 (4) |
| C2—C1—C3 | 122.2 (3) | C9—C8—H8 | 116.7 |
| C2—C1—Br1 | 119.3 (2) | C7—C8—H8 | 116.7 |
| C3—C1—Br1 | 118.5 (2) | C8—C9—H9A | 120.0 |
| N1—C2—C1 | 124.0 (3) | C8—C9—H9B | 120.0 |
| N1—C2—H2 | 118.0 | H9A—C9—H9B | 120.0 |
| C1—C2—H2 | 118.0 | N3—C10—C11 | 114.1 (3) |
| C4—C3—C1 | 114.4 (3) | N3—C10—H10A | 108.7 |
| C4—C3—H3 | 122.8 | C11—C10—H10A | 108.7 |
| C1—C3—H3 | 122.8 | N3—C10—H10B | 108.7 |
| C3—C4—N3 | 133.4 (3) | C11—C10—H10B | 108.7 |
| C3—C4—C5 | 119.3 (3) | H10A—C10—H10B | 107.6 |
| N3—C4—C5 | 107.3 (3) | C12—C11—C10 | 127.0 (4) |
| N1—C5—N2 | 125.5 (3) | C12—C11—H11 | 116.5 |
| N1—C5—C4 | 126.7 (3) | C10—C11—H11 | 116.5 |
| N2—C5—C4 | 107.8 (2) | C11—C12—H12A | 120.0 |
| O1—C6—N3 | 126.9 (3) | C11—C12—H12B | 120.0 |
| O1—C6—N2 | 126.4 (3) | H12A—C12—H12B | 120.0 |
| N3—C6—N2 | 106.6 (3) | | |
| | | | |
| C5—N1—C2—C1 | 1.8 (5) | N3—C4—C5—N1 | −179.7 (3) |
| C3—C1—C2—N1 | −1.2 (5) | C3—C4—C5—N2 | −179.1 (3) |
| Br1—C1—C2—N1 | −179.5 (3) | N3—C4—C5—N2 | 0.5 (3) |
| C2—C1—C3—C4 | 0.2 (4) | C4—N3—C6—O1 | −176.8 (4) |
| Br1—C1—C3—C4 | 178.6 (2) | C10—N3—C6—O1 | −7.0 (5) |
| C1—C3—C4—N3 | −179.4 (3) | C4—N3—C6—N2 | 0.7 (3) |
| C1—C3—C4—C5 | 0.0 (4) | C10—N3—C6—N2 | 170.5 (3) |
| C6—N3—C4—C3 | 178.7 (3) | C5—N2—C6—O1 | 177.2 (4) |
| C10—N3—C4—C3 | 9.2 (6) | C7—N2—C6—O1 | 1.4 (5) |
| C6—N3—C4—C5 | −0.7 (3) | C5—N2—C6—N3 | −0.4 (3) |
| C10—N3—C4—C5 | −170.3 (3) | C7—N2—C6—N3 | −176.1 (3) |
| C2—N1—C5—N2 | 178.2 (3) | C5—N2—C7—C8 | 81.6 (4) |
| C2—N1—C5—C4 | −1.6 (5) | C6—N2—C7—C8 | −103.5 (3) |
| C6—N2—C5—N1 | −179.9 (3) | N2—C7—C8—C9 | 2.7 (6) |
| C7—N2—C5—N1 | −4.3 (5) | C4—N3—C10—C11 | −77.2 (4) |
| C6—N2—C5—C4 | −0.1 (3) | C6—N3—C10—C11 | 114.7 (4) |

| | | | |
|---|---|---|---|
| C7—N2—C5—C4 | 175.5 (3) | N3—C10—C11—C12 | −3.3 (6) |
| C3—C4—C5—N1 | 0.8 (5) | | |