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2,6-Dichloro-3-nitropyridine

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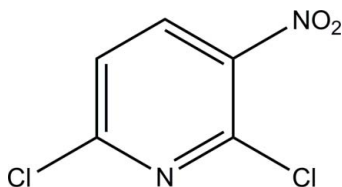
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.067; wR factor = 0.180; data-to-parameter ratio = 24.2.

The asymmetric unit of the title compound, $\text{C}_5\text{H}_2\text{Cl}_2\text{N}_2\text{O}_2$, consists of two crystallographically independent molecules. The pyridine ring in each molecule is essentially planar, with maximum deviations of 0.004 (4) and 0.007 (4) Å. Short $\text{Cl}\cdots\text{O}$ [3.09 (3) and 3.13 (4) Å] and $\text{Cl}\cdots\text{Cl}$ [3.38 (12) Å] contacts were observed. No significant intermolecular interactions were observed in the crystal packing.

Related literature

For the role of the nitropyridine nucleus in the development of medicinal agents and in the field of agrochemicals, see: Davis *et al.* (1996). For the properties and use of pyridine derivatives, see: Vacher *et al.* (1998); Olah *et al.* (1980); Bare *et al.* (1989). For standard bond lengths, see: Allen *et al.* (1987). For the melting point, see: Johnson *et al.* (1967).



Experimental

Crystal data

$\text{C}_5\text{H}_2\text{Cl}_2\text{N}_2\text{O}_2$
 $M_r = 192.99$

Monoclinic, $P2_1/c$
 $a = 7.9021$ (8) Å

$b = 19.166$ (2) Å
 $c = 11.0987$ (9) Å
 $\beta = 122.072$ (5)°
 $V = 1424.4$ (2) Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 0.85$ mm⁻¹
 $T = 296$ K
 $0.40 \times 0.27 \times 0.24$ mm

Data collection

Bruker SMART APEXII DUO
 CCD area-detector
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.727$, $T_{\max} = 0.821$

16845 measured reflections
 4817 independent reflections
 2323 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.180$
 $S = 1.08$
 4817 reflections

199 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.55$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.42$ e Å⁻³

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5183).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bare, T. M., McLaren, C. D., Campbell, D. J. B., Firor, J. W., Resch, J. F., Walters, C. P., Salama, A. I., Meiners, B. A. & Patel, J. B. (1989). *J. Med. Chem.* **32**, 2561–2573.
- Bruker (2009). *SADABS, APEX2 and SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Davis, L., Olsen, G. E., Klein, J. T., Kapples, K. J., Huger, F. P., Smith, C. P., Petko, W. W., Cornfeldt, M. & Efland, R. C. (1996). *J. Med. Chem.* **39**, 582–587.
- Johnson, C. D., Katritzky, A. R., Ridgewell, B. J. & Viney, M. (1967). *J. Chem. Soc. B*, pp. 1204–1210.
- Olah, G. A., Narang, S. C., Olah, J. A., Pearson, R. L. & Cupas, C. A. (1980). *J. Am. Chem. Soc.* **102**, 3507–3510.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Vacher, B., Bonnaud, B., Funes, P., Jubault, N., Koek, W., Assié, M.-B. & Cosi, C. (1998). *J. Med. Chem.* **41**, 5070–5083.

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supporting information

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2,6-Dichloro-3-nitropyridine

Hoong-Kun Fun, Suhana Arshad, B. Chandrakantha, Arun M. Isloor and Prakash Shetty

S1. Comment

Nitropyridine nucleus played a pivotal role in the development of different medicinal agents and in the field of agrochemicals (Davis *et al.*, 1996). It is seen from the current literature that pyridine derivatives have been developed and used as insecticidal agents (Vacher *et al.*, 1998). Nitrated pyridines and their derivatives are important intermediates in synthesis of heterocyclic compounds in dyes and pharmaceutical products (Olah *et al.*, 1980). Fused heterocycles containing nitropyridine systems have been associated with several biological and medicinal activities including antioxytic (Olah *et al.*, 1980), antiviral and anti-inflammatory (Bare *et al.*, 1989) profiles.

The asymmetric unit of the title compound (Fig. 1), consists of two crystallographically independent molecules *A* and *B*. The pyridine rings (N1/C1–C5) for molecules *A* and *B* are essentially planar with maximum deviations of 0.004 (4) Å at atom C1A and 0.007 (4) Å at atom C3B, respectively. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. In addition, short Cl \cdots O [Cl1A \cdots O2A (1 - *x*, -1/2 + *y*, 1/2 - *z*) = 3.093 (3) Å and Cl2A \cdots O2A (1 - *x*, 2 - *y*, -*z*) = 3.132 (4) Å] and Cl \cdots Cl [Cl2A \cdots Cl2A (1 - *x*, 2 - *y*, -*z*) = 3.3839 (12) Å] contacts were observed.

The crystal packing is shown in Fig. 2. No significant intermolecular interactions were observed in the crystal packing.

S2. Experimental

2,6-Dichloropyridine (5 g, 0.033 mol) was added lotwise to mixture of concentrated H₂SO₄ (25 ml) and fuming nitric acid (10 ml) at 0 °C. After the addition, the reaction mixture was heated to 65 °C for 2 h. After completion of the reaction, the reaction mixture was cooled to room temperature and quenched with ice water. The solid that separated out was filtered and dried under vacuum. The crude product was purified by column chromatography using silica gel 60–120 mesh size and petroleum ether: ethyl acetate as eluent to afford title compound as a pale yellow solid. Yield: 3.0 g, 46.0%. *M.p.*: 333–338 K (Johnson *et al.*, 1967).

S3. Refinement

All H atoms were positioned geometrically [C–H = 0.93 Å] and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. There is no pseudo-symmetry in the crystal structure.

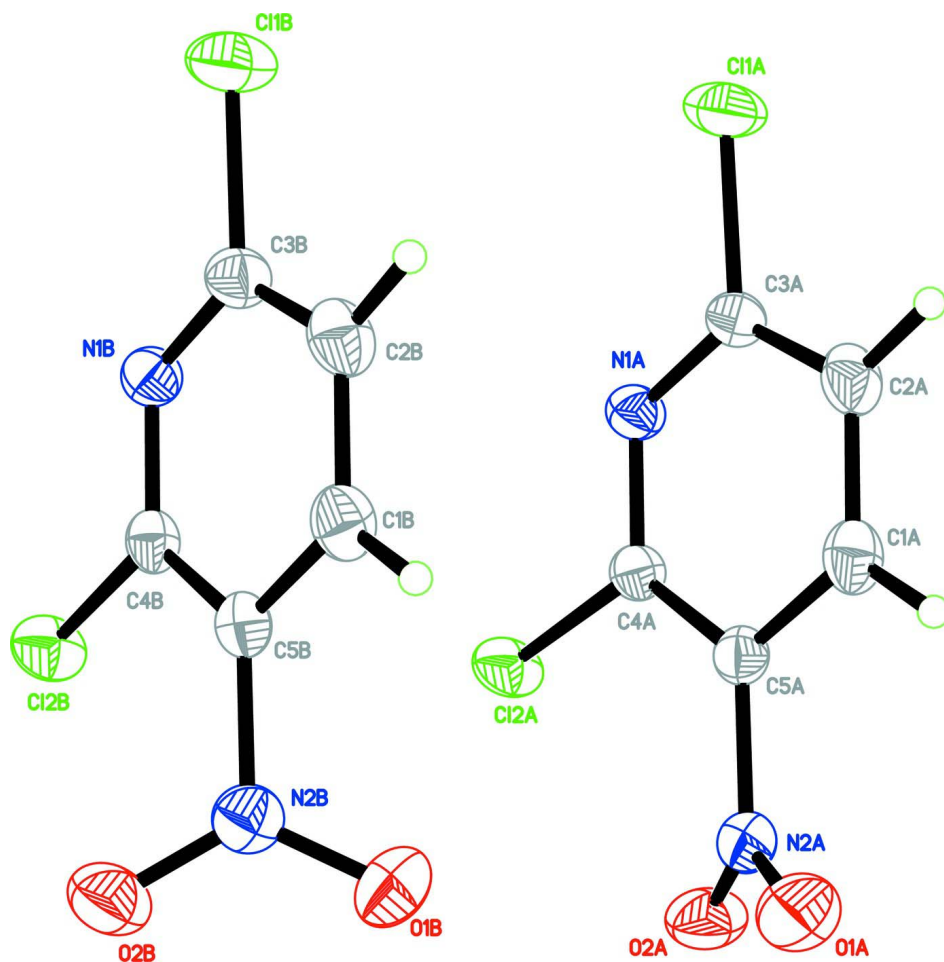


Figure 1

The molecular structure of the title compound, showing the two independent molecules with atom labels and 30% probability displacement ellipsoids.

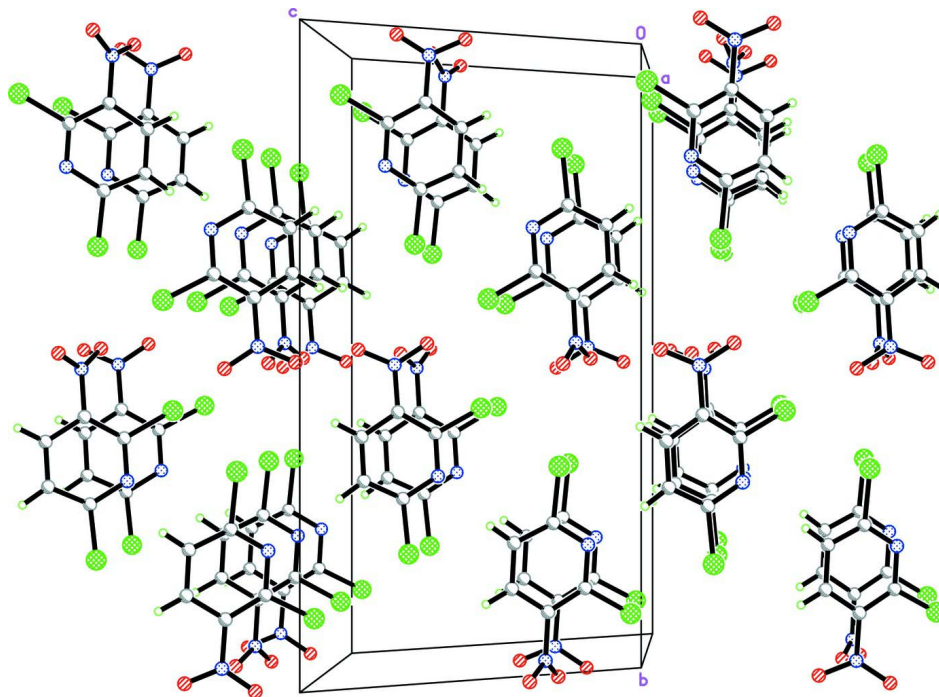


Figure 2

The crystal packing of the title compound.

2,6-Dichloro-3-nitropyridine

Crystal data

$C_5H_2Cl_2N_2O_2$

$M_r = 192.99$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 7.9021\ (8)\ \text{\AA}$

$b = 19.166\ (2)\ \text{\AA}$

$c = 11.0987\ (9)\ \text{\AA}$

$\beta = 122.072\ (5)^\circ$

$V = 1424.4\ (2)\ \text{\AA}^3$

$Z = 8$

$F(000) = 768$

$D_x = 1.800\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3419 reflections

$\theta = 2.4\text{--}31.7^\circ$

$\mu = 0.85\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, yellow

$0.40 \times 0.27 \times 0.24\ \text{mm}$

Data collection

Bruker SMART APEXII DUO CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)

$T_{\min} = 0.727$, $T_{\max} = 0.821$

16845 measured reflections

4817 independent reflections

2323 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.060$

$\theta_{\max} = 31.8^\circ$, $\theta_{\min} = 2.4^\circ$

$h = -11 \rightarrow 11$

$k = -28 \rightarrow 28$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.180$
 $S = 1.08$
 4817 reflections
 199 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
 $w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 1.2999P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.55 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1A	0.51344 (18)	0.68002 (5)	0.26085 (12)	0.0667 (3)
Cl2A	0.49627 (16)	0.91466 (4)	0.03753 (9)	0.0540 (3)
O1A	0.6135 (4)	1.00995 (14)	0.4256 (3)	0.0656 (8)
O2A	0.4472 (5)	1.01996 (13)	0.1975 (3)	0.0641 (8)
N1A	0.5042 (4)	0.80475 (13)	0.1709 (3)	0.0411 (6)
N2A	0.5284 (4)	0.98557 (14)	0.3053 (3)	0.0433 (6)
C1A	0.5404 (5)	0.87035 (18)	0.4058 (3)	0.0445 (8)
H1A	0.5538	0.8926	0.4848	0.053*
C2A	0.5356 (5)	0.79924 (18)	0.3979 (4)	0.0463 (8)
H2A	0.5436	0.7720	0.4701	0.056*
C3A	0.5182 (5)	0.76941 (15)	0.2786 (3)	0.0397 (7)
C4A	0.5072 (5)	0.87406 (15)	0.1788 (3)	0.0359 (7)
C5A	0.5251 (5)	0.90936 (15)	0.2945 (3)	0.0351 (7)
Cl1B	1.01800 (19)	0.67408 (5)	0.25606 (12)	0.0673 (3)
Cl2B	0.98563 (15)	0.91167 (5)	0.03234 (9)	0.0532 (3)
O1B	0.9156 (5)	1.00415 (15)	0.3366 (3)	0.0731 (9)
O2B	1.1216 (5)	1.01192 (14)	0.2658 (3)	0.0663 (8)
N1B	1.0071 (4)	0.79906 (13)	0.1667 (3)	0.0403 (6)
N2B	1.0196 (5)	0.97918 (15)	0.2975 (3)	0.0470 (7)
C1B	1.0342 (5)	0.86410 (18)	0.4000 (3)	0.0450 (8)
H1B	1.0422	0.8863	0.4773	0.054*
C2B	1.0342 (5)	0.79259 (19)	0.3928 (4)	0.0481 (8)
H2B	1.0421	0.7650	0.4646	0.058*
C3B	1.0218 (5)	0.76313 (16)	0.2741 (3)	0.0427 (8)
C4B	1.0086 (5)	0.86795 (16)	0.1754 (3)	0.0356 (7)

C5B 1.0219 (5) 0.90270 (16) 0.2895 (3) 0.0376 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11A	0.0972 (8)	0.0347 (4)	0.0768 (7)	0.0060 (5)	0.0521 (7)	0.0129 (4)
C12A	0.0868 (7)	0.0430 (4)	0.0416 (5)	-0.0036 (4)	0.0405 (5)	0.0053 (3)
O1A	0.085 (2)	0.0609 (17)	0.0551 (16)	-0.0130 (15)	0.0404 (16)	-0.0206 (13)
O2A	0.093 (2)	0.0406 (13)	0.0633 (17)	0.0096 (13)	0.0444 (17)	0.0094 (12)
N1A	0.0520 (18)	0.0343 (12)	0.0418 (15)	0.0003 (12)	0.0281 (14)	0.0016 (11)
N2A	0.0494 (17)	0.0377 (13)	0.0509 (17)	-0.0023 (12)	0.0321 (15)	-0.0046 (12)
C1A	0.049 (2)	0.056 (2)	0.0346 (17)	0.0028 (16)	0.0257 (17)	0.0035 (14)
C2A	0.050 (2)	0.055 (2)	0.0391 (18)	0.0067 (16)	0.0274 (17)	0.0137 (15)
C3A	0.0457 (19)	0.0331 (14)	0.0429 (18)	0.0047 (13)	0.0252 (17)	0.0086 (13)
C4A	0.0435 (19)	0.0332 (14)	0.0329 (16)	0.0010 (13)	0.0215 (15)	0.0025 (11)
C5A	0.0392 (18)	0.0337 (14)	0.0353 (16)	0.0015 (13)	0.0218 (14)	0.0012 (12)
C11B	0.0970 (9)	0.0390 (5)	0.0691 (7)	0.0050 (5)	0.0463 (6)	0.0105 (4)
C12B	0.0762 (7)	0.0501 (5)	0.0381 (4)	-0.0015 (4)	0.0335 (5)	0.0079 (3)
O1B	0.084 (2)	0.0648 (18)	0.093 (2)	0.0032 (15)	0.062 (2)	-0.0159 (16)
O2B	0.093 (2)	0.0517 (15)	0.0721 (18)	-0.0135 (15)	0.0556 (18)	-0.0038 (13)
N1B	0.0452 (16)	0.0392 (14)	0.0387 (15)	0.0028 (12)	0.0238 (13)	0.0050 (11)
N2B	0.0530 (18)	0.0447 (15)	0.0421 (16)	-0.0030 (14)	0.0244 (15)	-0.0050 (12)
C1B	0.051 (2)	0.0542 (19)	0.0355 (17)	-0.0033 (16)	0.0268 (17)	-0.0007 (14)
C2B	0.052 (2)	0.059 (2)	0.0369 (18)	-0.0006 (17)	0.0255 (17)	0.0104 (15)
C3B	0.046 (2)	0.0391 (16)	0.0417 (19)	0.0036 (14)	0.0224 (17)	0.0085 (14)
C4B	0.0360 (18)	0.0426 (16)	0.0296 (15)	0.0014 (13)	0.0183 (14)	0.0045 (12)
C5B	0.0385 (18)	0.0420 (16)	0.0358 (16)	-0.0011 (14)	0.0221 (15)	0.0013 (13)

Geometric parameters (Å, °)

C11A—C3A	1.723 (3)	C11B—C3B	1.717 (3)
C12A—C4A	1.711 (3)	C12B—C4B	1.717 (3)
O1A—N2A	1.224 (3)	O1B—N2B	1.214 (4)
O2A—N2A	1.209 (4)	O2B—N2B	1.211 (4)
N1A—C4A	1.331 (4)	N1B—C4B	1.323 (4)
N1A—C3A	1.326 (4)	N1B—C3B	1.327 (4)
N2A—C5A	1.465 (4)	N2B—C5B	1.469 (4)
C1A—C2A	1.365 (5)	C1B—C2B	1.373 (5)
C1A—C5A	1.393 (4)	C1B—C5B	1.391 (4)
C1A—H1A	0.9300	C1B—H1B	0.9300
C2A—C3A	1.381 (5)	C2B—C3B	1.389 (5)
C2A—H2A	0.9300	C2B—H2B	0.9300
C4A—C5A	1.391 (4)	C4B—C5B	1.385 (4)
C4A—N1A—C3A	117.4 (3)	C4B—N1B—C3B	117.3 (3)
O2A—N2A—O1A	124.5 (3)	O2B—N2B—O1B	125.5 (3)
O2A—N2A—C5A	119.0 (3)	O2B—N2B—C5B	117.9 (3)
O1A—N2A—C5A	116.5 (3)	O1B—N2B—C5B	116.6 (3)

C2A—C1A—C5A	119.5 (3)	C2B—C1B—C5B	118.8 (3)
C2A—C1A—H1A	120.2	C2B—C1B—H1B	120.6
C5A—C1A—H1A	120.2	C5B—C1B—H1B	120.6
C1A—C2A—C3A	117.4 (3)	C1B—C2B—C3B	117.3 (3)
C1A—C2A—H2A	121.3	C1B—C2B—H2B	121.4
C3A—C2A—H2A	121.3	C3B—C2B—H2B	121.4
N1A—C3A—C2A	124.8 (3)	N1B—C3B—C2B	124.7 (3)
N1A—C3A—C11A	114.8 (2)	N1B—C3B—C11B	115.0 (3)
C2A—C3A—C11A	120.4 (2)	C2B—C3B—C11B	120.2 (3)
N1A—C4A—C5A	122.4 (3)	N1B—C4B—C5B	122.7 (3)
N1A—C4A—C12A	113.8 (2)	N1B—C4B—C12B	115.3 (2)
C5A—C4A—C12A	123.8 (2)	C5B—C4B—C12B	122.0 (2)
C1A—C5A—C4A	118.4 (3)	C4B—C5B—C1B	119.1 (3)
C1A—C5A—N2A	118.2 (3)	C4B—C5B—N2B	122.5 (3)
C4A—C5A—N2A	123.3 (3)	C1B—C5B—N2B	118.4 (3)
C5A—C1A—C2A—C3A	0.9 (5)	C5B—C1B—C2B—C3B	0.0 (5)
C4A—N1A—C3A—C2A	0.0 (5)	C4B—N1B—C3B—C2B	1.5 (5)
C4A—N1A—C3A—C11A	-180.0 (3)	C4B—N1B—C3B—C11B	179.7 (2)
C1A—C2A—C3A—N1A	-0.6 (6)	C1B—C2B—C3B—N1B	-1.1 (6)
C1A—C2A—C3A—C11A	179.4 (3)	C1B—C2B—C3B—C11B	-179.2 (3)
C3A—N1A—C4A—C5A	0.3 (5)	C3B—N1B—C4B—C5B	-0.9 (5)
C3A—N1A—C4A—C12A	178.0 (2)	C3B—N1B—C4B—C12B	-179.1 (2)
C2A—C1A—C5A—C4A	-0.6 (5)	N1B—C4B—C5B—C1B	0.0 (5)
C2A—C1A—C5A—N2A	179.3 (3)	C12B—C4B—C5B—C1B	178.1 (3)
N1A—C4A—C5A—C1A	0.0 (5)	N1B—C4B—C5B—N2B	-178.8 (3)
C12A—C4A—C5A—C1A	-177.5 (3)	C12B—C4B—C5B—N2B	-0.7 (5)
N1A—C4A—C5A—N2A	-179.9 (3)	C2B—C1B—C5B—C4B	0.5 (5)
C12A—C4A—C5A—N2A	2.6 (5)	C2B—C1B—C5B—N2B	179.3 (3)
O2A—N2A—C5A—C1A	-153.8 (3)	O2B—N2B—C5B—C4B	-44.8 (5)
O1A—N2A—C5A—C1A	25.6 (4)	O1B—N2B—C5B—C4B	135.7 (4)
O2A—N2A—C5A—C4A	26.1 (5)	O2B—N2B—C5B—C1B	136.4 (3)
O1A—N2A—C5A—C4A	-154.5 (3)	O1B—N2B—C5B—C1B	-43.0 (5)