



# Crystal structure of 2,6-dichloro-4-nitropyridine *N*-oxide

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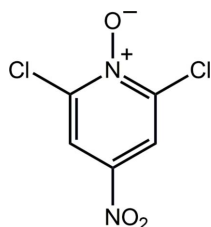
In the title compound,  $C_5H_2Cl_2N_2O_3$ , the nitro group is essentially coplanar with the aromatic ring, with a twist angle of  $4.00(6)^\circ$  and a fold angle of  $2.28(17)^\circ$ . The crystal structure exhibits a herringbone pattern with the zigzag running along the *b* axis. The herringbone layer-to-layer distance is  $3.0075(15)$  Å, with a shift of  $5.150(4)$  Å. Neighboring molecules are tilted at a  $57.83(4)^\circ$  (ring-to-ring) angle with each other. The nitro group on one molecule points to the *N*-oxide group on the neighboring one, with an intermolecular  $O \cdots N(\text{nitro})$  distance of  $3.1725(13)$  Å.

**Keywords:** crystal structure; pyridine *N*-oxide; herringbone pattern.

**CCDC reference:** 1425488

## 1. Related literature

For the synthesis of the title compound and related compounds, see: Rousseau & Robins (1965). For chemical interest in derivatives of pyridine *N*-oxide, including the ruthenium-catalyzed use of these compounds towards the epoxidation of olefins *via* an *N*-oxide coordinated  $Ru^{IV}=O$  intermediate, see: Gross & Ini (1999).



## 2. Experimental

### 2.1. Crystal data

$C_5H_2Cl_2N_2O_3$   
 $M_r = 208.99$   
Orthorhombic, *Pbca*  
 $a = 5.964(4)$  Å  
 $b = 9.510(6)$  Å  
 $c = 26.192(16)$  Å

$V = 1485.5(16)$  Å<sup>3</sup>  
 $Z = 8$   
Mo  $K\alpha$  radiation  
 $\mu = 0.84$  mm<sup>-1</sup>  
 $T = 173$  K  
 $0.6 \times 0.2 \times 0.1$  mm

### 2.2. Data collection

Rigaku XtaLAB mini  
diffractometer  
Absorption correction: multi-scan  
(*REQAB*; Rigaku, 1998)  
 $T_{\min} = 0.709$ ,  $T_{\max} = 1.000$

13695 measured reflections  
1697 independent reflections  
1512 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.043$

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.078$   
 $S = 1.09$   
1697 reflections

109 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.23$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.34$  e Å<sup>-3</sup>

Data collection: *CrystalClear-SM Expert* (Rigaku, 2011); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5385).

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## supporting information

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**Crystal structure of 2,6-dichloro-4-nitropyridine *N*-oxide**

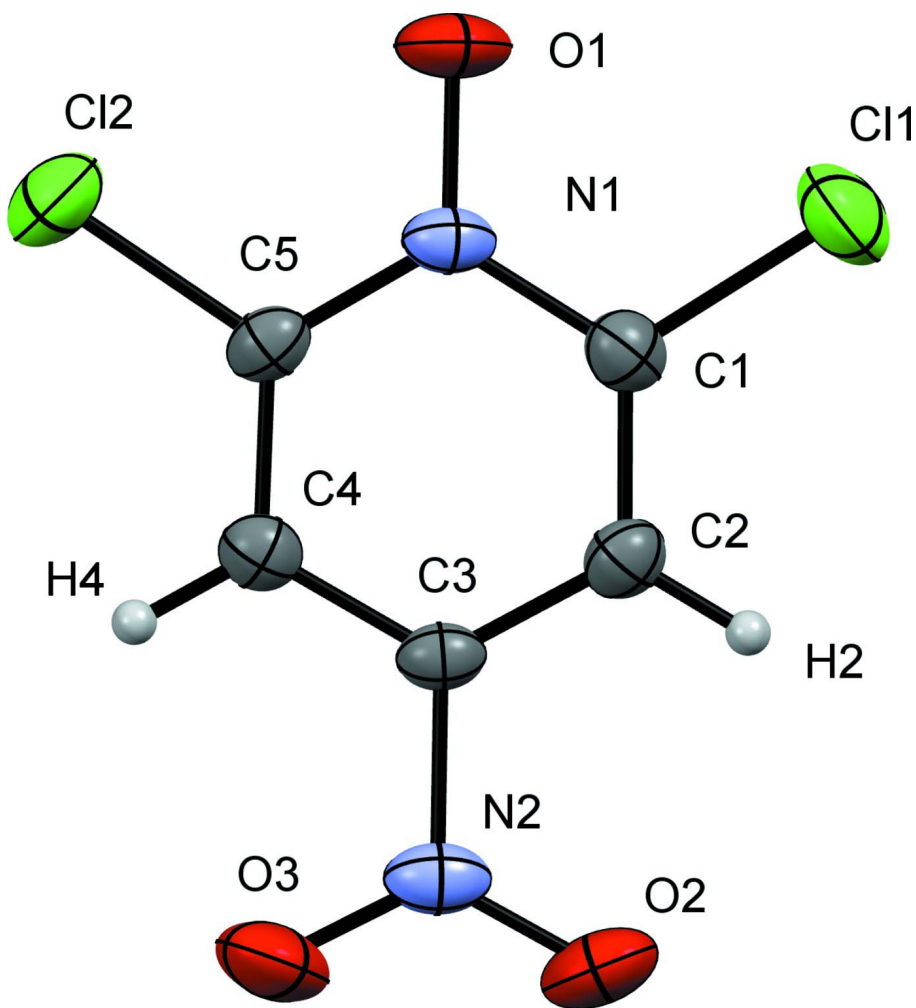
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**S1. Synthesis and crystallization**

2,6-Dichloro-4-nitropyridine *N*-oxide was purchased from Sigma-Aldrich and 0.10 g was dissolved in approximately 50 mL of methanol. Diffraction quality crystals were obtained by slow evaporation of the solvent.

**S2. Refinement**

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.95 Å) and were included in the refinement in the riding model approximation, with  $U_{\text{iso}}(\text{H})$  set to  $1.2U_{\text{equiv}}(\text{C})$ .

**Figure 1**

A view of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

### 2,6-Dichloro-4-nitropyridine *N*-oxide

#### Crystal data

$C_5H_2Cl_2N_2O_3$

$M_r = 208.99$

Orthorhombic, *Pbca*

$a = 5.964 (4) \text{ \AA}$

$b = 9.510 (6) \text{ \AA}$

$c = 26.192 (16) \text{ \AA}$

$V = 1485.5 (16) \text{ \AA}^3$

$Z = 8$

$F(000) = 832$

#### Data collection

Rigaku XtaLAB mini

diffractometer

Radiation source: Sealed Tube

$D_x = 1.869 \text{ Mg m}^{-3}$

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

Cell parameters from 3422 reflections

$\theta = 2.1\text{--}27.5^\circ$

$\mu = 0.84 \text{ mm}^{-1}$

$T = 173 \text{ K}$

Prism, colorless

$0.6 \times 0.2 \times 0.1 \text{ mm}$

Graphite Monochromator monochromator

Detector resolution:  $13.6612 \text{ pixels mm}^{-1}$

profile data from  $\omega$  scans

Absorption correction: multi-scan

(REQAB; Rigaku, 1998)

 $T_{\min} = 0.709$ ,  $T_{\max} = 1.000$ 

13695 measured reflections

1697 independent reflections

1512 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.043$  $\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.1^\circ$  $h = -7 \rightarrow 7$  $k = -12 \rightarrow 12$  $l = -33 \rightarrow 34$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.032$  $wR(F^2) = 0.078$  $S = 1.09$ 

1697 reflections

109 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methodsHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 0.628P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.34 \text{ e } \text{\AA}^{-3}$ *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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl2	0.85784 (8)	0.45634 (5)	0.29466 (2)	0.04219 (16)
Cl1	0.76404 (10)	0.58023 (6)	0.48666 (2)	0.05096 (18)
O1	0.9698 (2)	0.47138 (13)	0.39871 (5)	0.0406 (3)
O2	0.0755 (2)	0.79615 (14)	0.39133 (6)	0.0436 (3)
O3	0.1322 (2)	0.76147 (15)	0.31036 (6)	0.0494 (4)
N1	0.7830 (2)	0.53287 (14)	0.38874 (5)	0.0278 (3)
N2	0.1835 (2)	0.74825 (15)	0.35543 (6)	0.0337 (3)
C3	0.3887 (3)	0.67006 (16)	0.36721 (6)	0.0261 (3)
C4	0.5076 (3)	0.60983 (16)	0.32781 (6)	0.0273 (3)
H4	0.4563	0.6160	0.2935	0.033*
C2	0.4594 (3)	0.66162 (17)	0.41700 (7)	0.0297 (4)
H2	0.3734	0.7017	0.4439	0.036*
C5	0.7030 (3)	0.54043 (16)	0.33971 (6)	0.0268 (3)
C1	0.6574 (3)	0.59381 (17)	0.42689 (7)	0.0302 (4)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl2	0.0471 (3)	0.0357 (3)	0.0438 (3)	0.0101 (2)	0.0179 (2)	0.00214 (18)
Cl1	0.0628 (4)	0.0529 (3)	0.0372 (3)	0.0068 (2)	−0.0209 (2)	−0.0034 (2)
O1	0.0271 (6)	0.0318 (7)	0.0630 (9)	0.0093 (5)	−0.0097 (6)	0.0019 (6)
O2	0.0319 (7)	0.0348 (7)	0.0639 (9)	0.0093 (6)	0.0106 (6)	0.0024 (6)
O3	0.0438 (8)	0.0493 (9)	0.0552 (9)	0.0111 (7)	−0.0184 (7)	0.0051 (7)

N1	0.0229 (7)	0.0207 (7)	0.0397 (8)	0.0013 (5)	−0.0042 (6)	0.0013 (6)
N2	0.0260 (7)	0.0242 (7)	0.0508 (9)	0.0009 (6)	−0.0035 (7)	0.0047 (6)
C3	0.0223 (7)	0.0202 (7)	0.0357 (9)	0.0005 (6)	−0.0006 (6)	0.0026 (6)
C4	0.0293 (8)	0.0223 (8)	0.0303 (8)	−0.0013 (6)	−0.0016 (7)	0.0021 (6)
C2	0.0325 (9)	0.0238 (8)	0.0330 (9)	0.0021 (7)	0.0018 (7)	−0.0019 (7)
C5	0.0281 (8)	0.0204 (8)	0.0319 (9)	0.0006 (6)	0.0045 (7)	0.0012 (6)
C1	0.0341 (9)	0.0255 (8)	0.0310 (9)	−0.0005 (7)	−0.0050 (7)	−0.0006 (6)

*Geometric parameters (Å, °)*

Cl2—C5	1.6984 (18)	N2—C3	1.465 (2)
Cl1—C1	1.695 (2)	C3—C4	1.377 (2)
O1—N1	1.2847 (18)	C3—C2	1.373 (2)
O2—N2	1.228 (2)	C4—H4	0.9500
O3—N2	1.226 (2)	C4—C5	1.375 (2)
N1—C5	1.372 (2)	C2—H2	0.9500
N1—C1	1.377 (2)	C2—C1	1.370 (3)
O1—N1—C5	121.03 (14)	C5—C4—H4	121.1
O1—N1—C1	121.06 (15)	C3—C2—H2	120.9
C5—N1—C1	117.90 (14)	C1—C2—C3	118.13 (16)
O2—N2—C3	117.74 (15)	C1—C2—H2	120.9
O3—N2—O2	124.65 (16)	N1—C5—Cl2	115.89 (13)
O3—N2—C3	117.61 (15)	N1—C5—C4	122.15 (15)
C4—C3—N2	118.91 (15)	C4—C5—Cl2	121.96 (14)
C2—C3—N2	119.10 (15)	N1—C1—Cl1	115.73 (13)
C2—C3—C4	121.98 (16)	C2—C1—Cl1	122.26 (14)
C3—C4—H4	121.1	C2—C1—N1	122.01 (16)
C5—C4—C3	117.79 (16)		