Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

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

Andrew M. Prichard, Will E. Lynch and Clifford W. Padgett*

Armstrong State University, 11935 Abercorn St., Savanah, GA 31419, USA.
*Correspondence e-mail: clifford.padgett@armstrong.edu

Received 11 September 2015; accepted 17 September 2015

Edited by E. R. T. Tiekink, University of Malaya, Malaysia

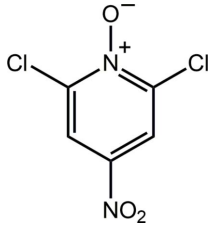
In the title compound, $C_5H_2Cl_2N_2O_3$, the nitro group is essentially coplanar with the aromatic ring, with a twist angle of 4.00 (6)° and a fold angle of 2.28 (17)°. The crystal structure exhibits a herringbone pattern with the zigzag running along the *b* axis. The herringbone layer-to-layer distance is 3.0075 (15) Å, with a shift of 5.150 (4) Å. Neighboring molecules are tilted at a 57.83 (4)° (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···N(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^3
Z = 8
Mo *Kα* radiation
μ = 0.84 mm^{-1}
T = 173 K
0.6 × 0.2 × 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_{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^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Acknowledgements

The authors acknowledge financial support from Armstrong State University.

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

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supporting information

Acta Cryst. (2015). E**71**, o775 [doi:10.1107/S2056989015017387]

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_{iso}(H) set to 1.2U_{equiv}(C).

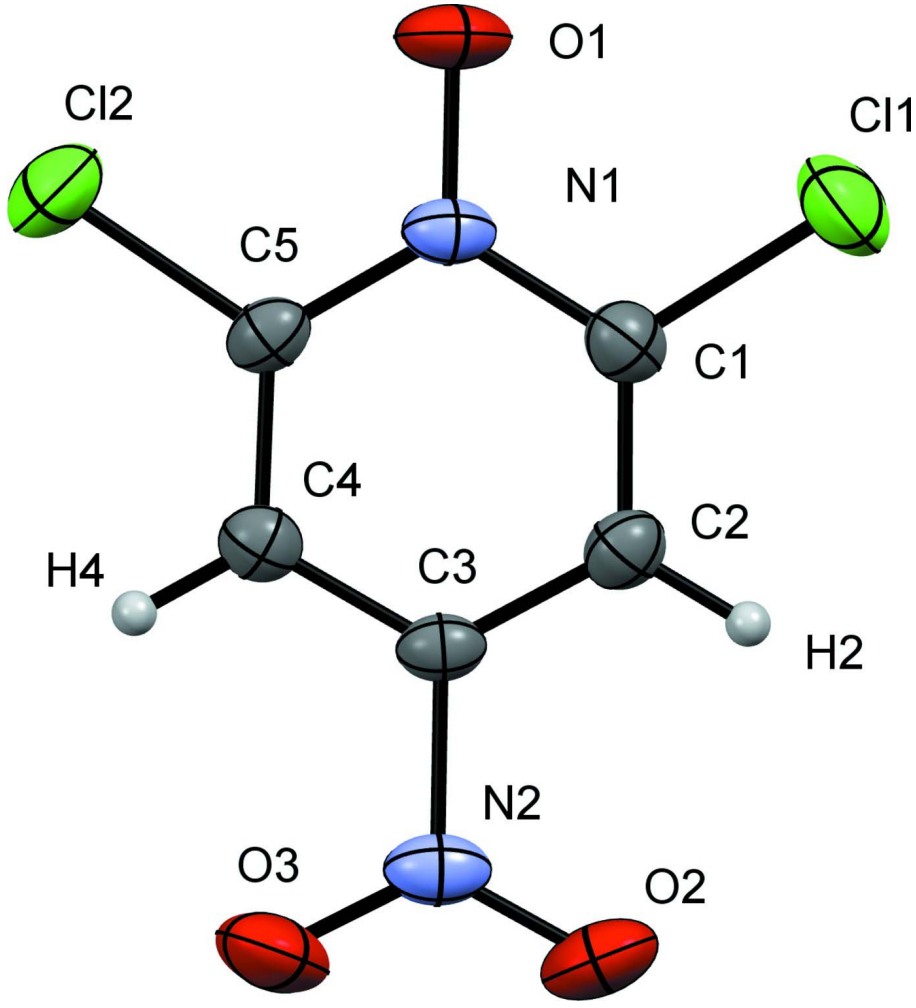

Figure 1

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) Å
b = 9.510 (6) Å
c = 26.192 (16) Å
V = 1485.5 (16) Å^3
Z = 8
F(000) = 832

D_x = 1.869 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3422 reflections
θ = 2.1–27.5°
μ = 0.84 mm^{-1}
T = 173 K
Prism, colorless
0.6 × 0.2 × 0.1 mm

Data collection

Rigaku XtaLAB mini diffractometer
Radiation source: Sealed Tube

Graphite Monochromator monochromator
Detector resolution: 13.6612 pixels mm^{-1}
profile data from ω 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_{int} = 0.043
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −7→7
k = −12→12
l = −33→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 methods
Hydrogen 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 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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, º)

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)		