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**Structural data**: full structural data are available from iucrdata.iucr.org

# 2-Chloro-4-nitropyridine *N*-oxide

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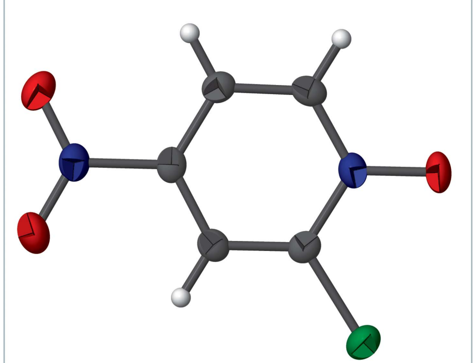
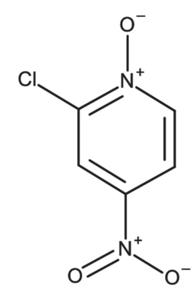
In the title compound, $C_5H_3ClN_2O_3$ (systematic name: 2-chloro-4-nitropyridin-1-ium-1-olate), the nitro group is essentially coplanar with the aromatic ring, with a twist angle of 6.48 (8)°. The molecular packing exhibits a herringbone pattern with the zigzag running along the *b* axis; here, there are no short contacts, hydrogen bonds, or $\pi$–$\pi$ interactions.

**3D view**



**Chemical scheme**




## Structure description

Pyridine *N*-oxide and related compounds have garnered much interest in organic chemistry since their preparation was first reported by Meisenheimer (1926). A number of recent publications have highlighted their utility in organic transformations such as reactions with Grignard reagents (Andersson *et al.*, 2011), aromatic ring substitutions (Shibata & Takano, 2015) and aromatic coupling reactions (Wang & Zhang, 2015). Further, numerous uses in pharmaceutical applications have been realised throughout the years, such as the recent report of uses as an emerging class of therapeutic agents, including thrombin as a potential clotting inhibitor drug (Mfuh & Larionov, 2015).

In the title compound (Fig. 1), the nitro group is essentially coplanar with the aromatic ring, with a twist angle of 6.48 (8)°. The crystal structure (Fig. 2) exhibits a herringbone pattern with the zigzag running along the *b* axis. The herringbone layer-to-layer distance is 2.947 (4) Å with a shift of 5.155 (5) Å. Neighboring molecules of the herringbone are tilted at a 47.08 (10)° (ring-to-ring) angle to each other. The chloro group in one of herringbone chains points to the chloro group in the neighboring one, with a Cl$\cdots$Cl intermolecular distance of 3.708 (2) Å. In the bends of the chains, the *N*-oxide aligns with the nitro group with an O$\cdots$O distance of 2.922 (3) Å. There are no other short contacts, hydrogen bonds, or $\pi$–$\pi$ interactions.

This structure is similar to the previously reported structure of 2,6-dichloro-4-nitropyridine *N*-oxide (Prichard *et al.*, 2015).

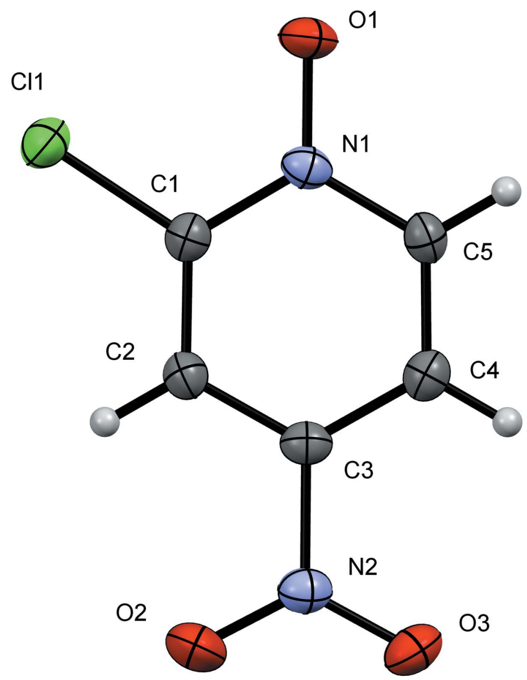



**Figure 1**
A view of the molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

**Table 1**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_5H_3ClN_2O_3$ |
| $M_r$ | 174.54 |
| Crystal system, space group | Orthorhombic, *Pbca* |
| Temperature (K) | 176 |
| $a$, $b$, $c$ (Å) | 5.9238 (14), 9.735 (2), 22.444 (8) |
| $V$ (Å$^3$) | 1294.3 (5) |
| $Z$ | 8 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.54 |
| Crystal size (mm) | 0.34 × 0.18 × 0.08 |
| Data collection | |
| Diffractometer | Rigaku XtalLab mini CCD |
| Absorption correction | Multi-scan (*REQAB*;Rigaku, 1998) |
| $T_{min}$, $T_{max}$ | 0.741, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 11029, 1485, 1088 |
| $R_{int}$ | 0.111 |
| $(\sin \theta/\lambda)_{max}$ (Å$^{-1}$) | 0.649 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.050, 0.132, 1.06 |
| No. of reflections | 1484 |
| No. of parameters | 100 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.33, −0.33 |

Computer programs: *CrystalClear* (Rigaku, 2009), *SHELXT* (Sheldrick, 2015*b*), *SHELXL* (Sheldrick, 2015*a*) and *OLEX2* (Dolomanov *et al.*, 2009).

## Synthesis and crystallization

2-Chloro-4-nitropyridine *N*-oxide was purchased from Sigma–Aldrich and 0.10 g was dissolved in approximately 50 ml of chloroform. Diffraction-quality crystals were obtained by slow evaporation of the solvent.

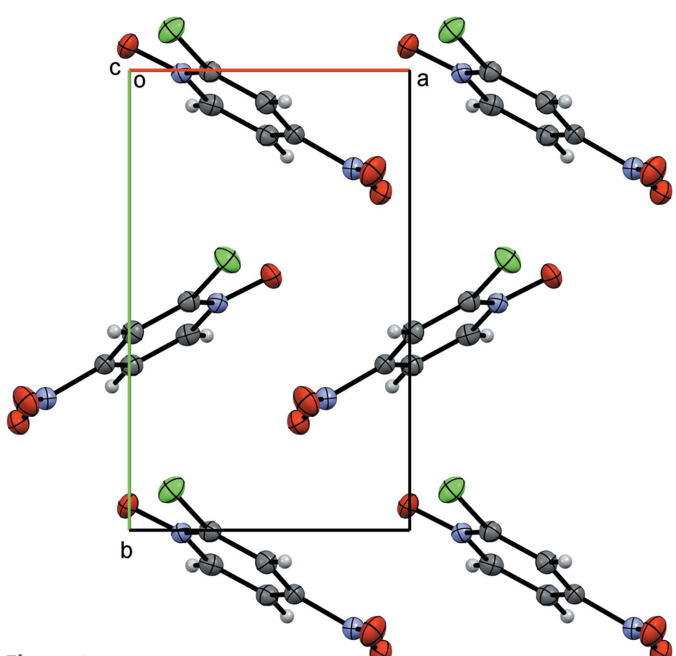



**Figure 2**
Molecular packing diagram of title compound viewed along the *c* axis.

## Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 1.

## Funding information

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# full crystallographic data

*IUCrData* (2018). **3**, x180016 [https://doi.org/10.1107/S2414314618000160]

## 2-Chloro-4-nitropyridine *N*-oxide

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### 2-Chloro-4-nitropyridine *N*-oxide

*Crystal data*

$C_5H_3ClN_2O_3$
$M_r$ = 174.54
Orthorhombic, *Pbca*
*a* = 5.9238 (14) Å
*b* = 9.735 (2) Å
*c* = 22.444 (8) Å
*V* = 1294.3 (5) Å$^3$
*Z* = 8
*F*(000) = 704
$D_x$ = 1.791 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2672 reflections
*θ* = 2.1–27.5°
*μ* = 0.54 mm$^{-1}$
*T* = 176 K
Prism, colorless
0.34 × 0.18 × 0.08 mm

*Data collection*

Rigaku XtalLab mini CCD diffractometer
*ω* scans
Absorption correction: multi-scan (*REQAB*;Rigaku, 1998)
$T_{min}$ = 0.741, $T_{max}$ = 1.000
11029 measured reflections
1485 independent reflections
1088 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.111
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.8°
*h* = −7→7
*k* = −12→12
*l* = −28→28

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.132
*S* = 1.06
1484 reflections
100 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.245P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.33 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**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 1.2$U_{equiv}$(C)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.84970 (12) | 0.41241 (8) | 0.70078 (3) | 0.0328 (3) |
| O1 | 1.0061 (3) | 0.4471 (2) | 0.58347 (8) | 0.0308 (5) |
| O2 | 0.1310 (3) | 0.7204 (2) | 0.67343 (9) | 0.0394 (6) |
| O3 | 0.1031 (3) | 0.7656 (2) | 0.57923 (8) | 0.0327 (5) |
| N1 | 0.8139 (3) | 0.5062 (2) | 0.59237 (9) | 0.0230 (5) |
| N2 | 0.2001 (4) | 0.7138 (2) | 0.62205 (9) | 0.0272 (5) |
| C1 | 0.7126 (4) | 0.5030 (3) | 0.64730 (10) | 0.0241 (6) |
| C2 | 0.5117 (4) | 0.5703 (3) | 0.65747 (11) | 0.0249 (6) |
| H2 | 0.445346 | 0.569168 | 0.695020 | 0.030* |
| C3 | 0.4116 (4) | 0.6391 (3) | 0.61119 (11) | 0.0244 (6) |
| C4 | 0.5083 (4) | 0.6418 (3) | 0.55541 (11) | 0.0252 (6) |
| H4 | 0.438517 | 0.688058 | 0.524175 | 0.030* |
| C5 | 0.7094 (4) | 0.5752 (3) | 0.54681 (11) | 0.0257 (6) |
| H5 | 0.776304 | 0.576716 | 0.509329 | 0.031* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0334 (4) | 0.0350 (4) | 0.0301 (4) | 0.0086 (3) | −0.0052 (3) | 0.0017 (3) |
| O1 | 0.0223 (10) | 0.0284 (11) | 0.0418 (12) | 0.0070 (9) | 0.0068 (8) | −0.0018 (8) |
| O2 | 0.0331 (12) | 0.0453 (14) | 0.0397 (12) | 0.0094 (10) | 0.0090 (9) | −0.0013 (10) |
| O3 | 0.0255 (10) | 0.0300 (11) | 0.0425 (12) | 0.0013 (9) | −0.0069 (8) | 0.0048 (9) |
| N1 | 0.0210 (12) | 0.0174 (11) | 0.0307 (11) | −0.0014 (10) | 0.0032 (8) | −0.0024 (9) |
| N2 | 0.0209 (12) | 0.0248 (12) | 0.0360 (13) | −0.0017 (10) | 0.0012 (9) | −0.0006 (10) |
| C1 | 0.0261 (14) | 0.0212 (13) | 0.0249 (12) | −0.0009 (12) | −0.0037 (10) | −0.0013 (10) |
| C2 | 0.0235 (14) | 0.0258 (14) | 0.0255 (13) | −0.0027 (12) | 0.0021 (10) | −0.0012 (10) |
| C3 | 0.0198 (13) | 0.0225 (13) | 0.0308 (14) | −0.0016 (11) | −0.0006 (11) | −0.0030 (10) |
| C4 | 0.0253 (14) | 0.0247 (14) | 0.0257 (14) | −0.0017 (11) | −0.0033 (10) | 0.0017 (10) |
| C5 | 0.0279 (14) | 0.0269 (14) | 0.0224 (13) | −0.0050 (12) | 0.0026 (10) | 0.0003 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C1 | 1.697 (3) | C1—C2 | 1.378 (4) |
| O1—N1 | 1.291 (3) | C2—H2 | 0.9300 |
| O2—N2 | 1.225 (3) | C2—C3 | 1.371 (4) |
| O3—N2 | 1.228 (3) | C3—C4 | 1.377 (4) |
| N1—C1 | 1.372 (4) | C4—H4 | 0.9300 |
| N1—C5 | 1.371 (3) | C4—C5 | 1.370 (4) |
| N2—C3 | 1.469 (4) | C5—H5 | 0.9300 |
| | | | |
| O1—N1—C1 | 121.0 (2) | C3—C2—H2 | 120.6 |
| O1—N1—C5 | 120.1 (2) | C2—C3—N2 | 119.0 (2) |
| C5—N1—C1 | 118.9 (2) | C2—C3—C4 | 121.2 (3) |
| O2—N2—O3 | 124.0 (3) | C4—C3—N2 | 119.7 (2) |
| O2—N2—C3 | 117.9 (2) | C3—C4—H4 | 120.6 |

| | | | |
|---|---|---|---|
| O3—N2—C3 | 118.2 (2) | C5—C4—C3 | 118.7 (2) |
| N1—C1—Cl1 | 116.0 (2) | C5—C4—H4 | 120.6 |
| N1—C1—C2 | 121.1 (2) | N1—C5—H5 | 119.4 |
| C2—C1—Cl1 | 122.9 (2) | C4—C5—N1 | 121.3 (2) |
| C1—C2—H2 | 120.6 | C4—C5—H5 | 119.4 |
| C3—C2—C1 | 118.7 (2) | | |