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3-Chloro-5-methoxy-2,6-dinitropyridine

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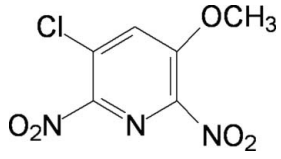
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.135; data-to-parameter ratio = 14.9.

In the crystal structure of the title compound, $C_6H_4ClN_3O_5$, the two nitro groups are twisted with respect to the pyridine ring, making dihedral angles of 33.12 (13) and 63.66 (14)°.

Related literature

For the synthesis, see: Bissell & Swansiger (1987); Chen *et al.* (2008).



Experimental

Crystal data

$C_6H_4ClN_3O_5$ M_r = 233.57
Monoclinic, $P2_1/n$
a = 6.6490 (13) Å
b = 10.842 (2) Å
c = 12.715 (3) Å
β = 95.55 (3)°
V = 912.3 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 293 K
0.50 × 0.40 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.808, T_{max} = 0.887
5866 measured reflections
2062 independent reflections
1275 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.135
S = 0.99
2062 reflections
138 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2586).

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supplementary materials

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3-Chloro-5-methoxy-2,6-dinitropyridine

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Comment

Pyridine derivatives are important intermediates used to synthesize pesticide, medicine and play important roles in fine chemical field. 3-Chloro-5-methoxyl- 2,6-dinitro-pyridine was synthesized from 3,5-dichloropyridine N-oxide by substitution and nitration (Bissell *et al.*, 1987), and the process was improved by Chen *et al.* (2008). The crystal structure of the title compound is presented here.

The molecular structure of the title compound is shown in Fig. 1. While the methoxyl group, except H atoms, is co-planar with the pyridine ring, the two nitro groups are twisted with respect to the pyridine ring with dihedral angles of 33.12 (13) and 63.66 (14)°, respectively. Neither hydrogen bonding nor π-π stacking is observed in the crystal structure.

Experimental

The title compound was prepared according to a literature method (Chen *et al.*, 2008). Crystals suitable for X-ray analysis were obtained by slow evaporation of 1,2-dichloroethane.

Refinement

H atoms were positioned geometrically and refined using a ride model with C—H = 0.93 Å for aromatic H and 0.96 Å for methyl H atoms, $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for aromatic H atom.

Figures

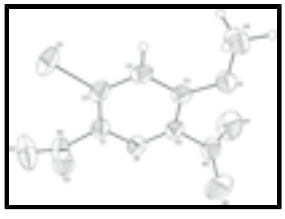

Fig. 1. The molecular structure of title compound, with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

3-Chloro-5-methoxy-2,6-dinitropyridine

Crystal data

$C_6H_4ClN_3O_5$	$F_{000} = 472$
$M_r = 233.57$	$D_x = 1.701$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 5866 reflections
$a = 6.6490$ (13) Å	$\theta = 2.5$–27.5º

b = 10.842 (2) Å	μ = 0.43 mm^{-1}
c = 12.715 (3) Å	T = 293 K
β = 95.55 (3)º	Block, colorless
V = 912.3 (3) Å^3	0.50 × 0.40 × 0.28 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	2062 independent reflections
Radiation source: fine-focus sealed tube	1275 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.050
Detector resolution: 10.00 pixels mm^{-1}	θ_{max} = 27.5º
T = 293 K	θ_{min} = 2.5º
ω scans	h = −8→8
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	k = −13→13
T_{min} = 0.808, T_{max} = 0.887	l = −16→16
5866 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.048	$w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.135	$(\Delta/\sigma)_{max} < 0.001$
S = 0.99	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
2062 reflections	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
138 parameters	Extinction correction: SHELXTL (Version 4.2; Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.157 (11)
Secondary atom site location: difference Fourier map	

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.17055 (10)	0.52802 (8)	0.42365 (6)	0.0756 (3)
O1	0.8010 (3)	0.77739 (16)	0.37270 (12)	0.0536 (5)
O2	0.2241 (4)	0.3362 (2)	0.27062 (18)	0.0939 (8)
O3	0.2590 (3)	0.4071 (2)	0.11455 (16)	0.0716 (6)
O4	0.8115 (4)	0.7839 (2)	0.14363 (19)	0.0949 (8)
O5	0.9482 (4)	0.6061 (2)	0.1524 (2)	0.1051 (9)
N1	0.5510 (3)	0.55114 (17)	0.20338 (15)	0.0439 (5)
N2	0.2845 (3)	0.4126 (2)	0.2107 (2)	0.0590 (6)
N3	0.8230 (3)	0.6789 (2)	0.17477 (16)	0.0556 (6)
C1	0.4068 (3)	0.5141 (2)	0.25985 (18)	0.0440 (5)
C2	0.3749 (3)	0.5646 (2)	0.35677 (18)	0.0453 (6)
C3	0.5061 (3)	0.6544 (2)	0.39881 (17)	0.0443 (5)
H3	0.4898	0.6885	0.4645	0.053*
C4	0.6614 (3)	0.6929 (2)	0.34246 (16)	0.0407 (5)
C5	0.6705 (3)	0.6373 (2)	0.24397 (16)	0.0412 (5)
C6	0.7921 (5)	0.8349 (3)	0.47424 (19)	0.0657 (8)
H6A	0.6641	0.8754	0.4761	0.099*
H6B	0.8989	0.8944	0.4857	0.099*
H6C	0.8073	0.7732	0.5286	0.099*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0570 (4)	0.0974 (6)	0.0777 (5)	−0.0107 (4)	0.0343 (4)	0.0139 (4)
O1	0.0575 (10)	0.0580 (10)	0.0474 (9)	−0.0144 (8)	0.0153 (8)	−0.0112 (8)
O2	0.1109 (18)	0.0831 (15)	0.0882 (15)	−0.0542 (15)	0.0115 (13)	0.0088 (12)
O3	0.0704 (12)	0.0772 (13)	0.0661 (13)	−0.0183 (10)	0.0006 (10)	−0.0065 (10)
O4	0.1047 (18)	0.0908 (18)	0.0946 (16)	−0.0134 (14)	0.0377 (14)	0.0320 (13)
O5	0.0869 (16)	0.1075 (19)	0.133 (2)	0.0047 (15)	0.0717 (15)	−0.0076 (16)
N1	0.0418 (10)	0.0451 (10)	0.0457 (10)	−0.0002 (9)	0.0083 (8)	−0.0001 (8)
N2	0.0494 (12)	0.0609 (14)	0.0666 (15)	−0.0129 (11)	0.0060 (10)	0.0026 (11)
N3	0.0526 (12)	0.0688 (15)	0.0481 (11)	−0.0127 (11)	0.0182 (9)	−0.0061 (11)
C1	0.0400 (11)	0.0430 (12)	0.0492 (12)	−0.0024 (10)	0.0053 (10)	0.0060 (10)
C2	0.0388 (11)	0.0492 (13)	0.0493 (13)	0.0041 (10)	0.0121 (9)	0.0141 (10)
C3	0.0468 (12)	0.0476 (13)	0.0405 (12)	0.0064 (11)	0.0150 (9)	0.0035 (10)
C4	0.0415 (11)	0.0395 (11)	0.0422 (12)	0.0038 (10)	0.0096 (9)	0.0035 (9)
C5	0.0364 (10)	0.0460 (12)	0.0423 (11)	0.0004 (10)	0.0097 (9)	0.0037 (9)
C6	0.0845 (19)	0.0678 (17)	0.0463 (14)	−0.0182 (15)	0.0138 (13)	−0.0125 (12)

Geometric parameters (Å, °)

Cl1—C2	1.717 (2)	N3—C5	1.476 (3)
O1—C4	1.334 (3)	C1—C2	1.384 (3)
O1—C6	1.440 (3)	C2—C3	1.380 (3)
O2—N2	1.219 (3)	C3—C4	1.378 (3)

O3—N2	1.219 (3)	C3—H3	0.9300
O4—N3	1.206 (3)	C4—C5	1.396 (3)
O5—N3	1.201 (3)	C6—H6A	0.9600
N1—C5	1.300 (3)	C6—H6B	0.9600
N1—C1	1.315 (3)	C6—H6C	0.9600
N2—C1	1.472 (3)		
C4—O1—C6	117.90 (18)	C4—C3—H3	120.3
C5—N1—C1	116.89 (19)	C2—C3—H3	120.3
O3—N2—O2	124.9 (2)	O1—C4—C3	126.4 (2)
O3—N2—C1	118.7 (2)	O1—C4—C5	117.78 (18)
O2—N2—C1	116.4 (2)	C3—C4—C5	115.8 (2)
O5—N3—O4	124.5 (2)	N1—C5—C4	126.05 (19)
O5—N3—C5	118.2 (2)	N1—C5—N3	114.31 (18)
O4—N3—C5	117.3 (2)	C4—C5—N3	119.6 (2)
N1—C1—C2	123.3 (2)	O1—C6—H6A	109.5
N1—C1—N2	113.5 (2)	O1—C6—H6B	109.5
C2—C1—N2	123.2 (2)	H6A—C6—H6B	109.5
C3—C2—C1	118.6 (2)	O1—C6—H6C	109.5
C3—C2—Cl1	118.16 (18)	H6A—C6—H6C	109.5
C1—C2—Cl1	123.13 (19)	H6B—C6—H6C	109.5
C4—C3—C2	119.3 (2)		
C5—N1—C1—C2	1.9 (3)	C6—O1—C4—C5	−180.0 (2)
C5—N1—C1—N2	−177.3 (2)	C2—C3—C4—O1	−179.3 (2)
O3—N2—C1—N1	−31.5 (3)	C2—C3—C4—C5	1.0 (3)
O2—N2—C1—N1	145.0 (2)	C1—N1—C5—C4	0.9 (3)
O3—N2—C1—C2	149.3 (2)	C1—N1—C5—N3	−177.3 (2)
O2—N2—C1—C2	−34.2 (4)	O1—C4—C5—N1	178.0 (2)
N1—C1—C2—C3	−3.1 (3)	C3—C4—C5—N1	−2.3 (3)
N2—C1—C2—C3	176.0 (2)	O1—C4—C5—N3	−4.0 (3)
N1—C1—C2—Cl1	172.71 (17)	C3—C4—C5—N3	175.7 (2)
N2—C1—C2—Cl1	−8.2 (3)	O5—N3—C5—N1	−64.2 (3)
C1—C2—C3—C4	1.4 (3)	O4—N3—C5—N1	115.0 (3)
Cl1—C2—C3—C4	−174.58 (16)	O5—N3—C5—C4	117.5 (3)
C6—O1—C4—C3	0.3 (3)	O4—N3—C5—C4	−63.3 (3)

Fig. 1