

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

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

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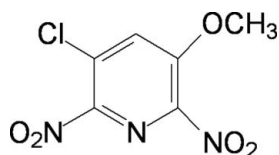
Received 8 August 2009; accepted 10 August 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.135; data-to-parameter ratio = 14.9.

In the crystal structure of the title compound, $\text{C}_6\text{H}_4\text{ClN}_3\text{O}_5$, 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

 $\text{C}_6\text{H}_4\text{ClN}_3\text{O}_5$ $M_r = 233.57$

Monoclinic, $P2_1/n$
 $a = 6.6490$ (13) Å
 $b = 10.842$ (2) Å
 $c = 12.715$ (3) Å
 $\beta = 95.55$ (3)°
 $V = 912.3$ (3) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.43$ mm⁻¹
 $T = 293$ K
 $0.50 \times 0.40 \times 0.28$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$

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/MS, 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*.

This work was supported by China North Industries Group Corporation (project No. ZC060302-15).

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

References

- Bissell, E. R. & Swansiger, R. W. (1987). *J. Heterocycl. Chem.* **24**, 59–62.
 Chen, J., Li, Q.-L. & Wang, J.-L. (2008). *Chin. J. Org. Chem.* **28**, 123–126.
 Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
 Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
 Rigaku (2000). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
 Rigaku/MS (2002). *CrystalStructure*. Rigaku/MS, The Woodlands, Texas, USA.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2009). E65, o2154 [doi:10.1107/S1600536809031407]

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-methoxy-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, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for aromatic H atom.

Figures

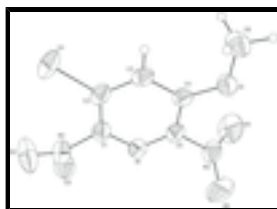


Fig. 1. The molecular structure of title compound, with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

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

Crystal data

$\text{C}_6\text{H}_4\text{ClN}_3\text{O}_5$

$M_r = 233.57$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 6.6490(13)$ Å

$F_{000} = 472$

$D_x = 1.701$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5866 reflections

$\theta = 2.5$ – 27.5°

supplementary materials

$b = 10.842(2) \text{ \AA}$
 $c = 12.715(3) \text{ \AA}$
 $\beta = 95.55(3)^\circ$
 $V = 912.3(3) \text{ \AA}^3$
 $Z = 4$

$\mu = 0.43 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
Block, colorless
 $0.50 \times 0.40 \times 0.28 \text{ mm}$

Data collection

Rigaku R-Axis RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
Detector resolution: $10.00 \text{ pixels mm}^{-1}$
 $T = 293 \text{ K}$
 ω scans
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.808$, $T_{\max} = 0.887$
5866 measured reflections

2062 independent reflections
1275 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$
 $\theta_{\text{max}} = 27.5^\circ$
 $\theta_{\text{min}} = 2.5^\circ$
 $h = -8 \rightarrow 8$
 $k = -13 \rightarrow 13$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.135$
 $S = 0.99$
2062 reflections
138 parameters

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
Extinction correction: SHELXTL (Version 4.2; Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x Fc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.157 (11)

Primary atom site location: structure-invariant direct methods
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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| 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 (\AA^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 (\AA , $^\circ$)

| | | | |
|--------|-----------|-------|-----------|
| 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) |

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| | | | |
|--------------|--------------|-------------|-------------|
| 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—C11 | 118.16 (18) | H6A—C6—H6C | 109.5 |
| C1—C2—C11 | 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—C11 | 172.71 (17) | C3—C4—C5—N3 | 175.7 (2) |
| N2—C1—C2—C11 | -8.2 (3) | O5—N3—C5—N1 | -64.2 (3) |
| C1—C2—C3—C4 | 1.4 (3) | O4—N3—C5—N1 | 115.0 (3) |
| C11—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

