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1,5-Dichloro-4,8-dinitroanthraquinone

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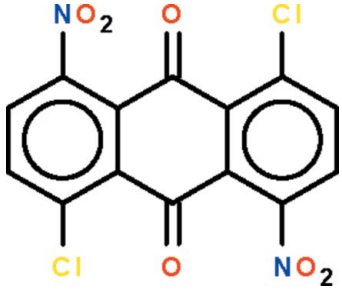
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.095; data-to-parameter ratio = 13.9.

The ring skeleton of the title compound, $C_{14}H_4Cl_2N_2O_6$, is close to planar (r.m.s. deviation of the carbon atoms 0.091 Å); the nitro goups are twisted with respect to the mean plane of the ring system by 70.8 (1) and 86.7 (2)°. The crystal studied was found to be a merohedral twin, with a domain ratio of 0.61 (8):0.39 (8).

Related literature

For dehydrosulfurization by using anthraquinone-based catalysts, see: Nagai *et al.* (1993). For a related structure, see: Armaghan *et al.* (2010).



Experimental

Crystal data

$C_{14}H_4Cl_2N_2O_6$
M_r = 367.09
Monoclinic, $P2_1$
a = 5.9596 (6) Å
b = 11.3897 (11) Å
c = 9.8667 (9) Å
β = 93.519 (1)°
V = 668.47 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.53 mm^{-1}
T = 100 K
0.12 × 0.12 × 0.12 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*, Sheldrick, 1996)
T_{min} = 0.940, T_{max} = 0.940
6510 measured reflections
3028 independent reflections
2779 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.095
S = 1.04
3028 reflections
218 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Absolute structure: Flack (Flack, 1983), 1402 Friedel pairs
Flack parameter: 0.39 (8)

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Shahid Beheshti University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2270).

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supplementary materials

Acta Cryst. (2010). E**66**, o2130 [**doi:10.1107/S1600536810029156**]

1,5-Dichloro-4,8-dinitroanthraquinone

M. Armaghan, M. M. Amini and S. W. Ng

Comment

The title compound (Scheme I, Fig. 1) belongs to a class of catalysts used for dehydrosulfurisation (Nagai *et al.*, 1993). We have embarked on a study of dehydrosulfurisation, and have recently reported the crysal structure of 1,8-dihydroxy-2,4,5,7-tetranitro-9,10-anthraquinone (Armaghan *et al.*, 2010). These compounds are synthesised by the reaction of fuming nitric acid on the substituted anthraquinone.

Experimental

Fuming nitric acid (10 ml) was added dropwise to a solution of 1,5-dichloroanthraquinone (277 mg, 1 mmol) in concentrated sulfuric acid (5 ml). The mixture was kept at 333 K. After two hours, the mixture was poured into ice (100 g). The organic compound was collected and dried. Crystals suitable for X-ray analysis were obtained by recrystallisation from toluene; m.p. > 540 K.

Refinement

Hydrogen atoms were placed in calculated positions (C–H 0.95 Å) and included in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C).

Figures

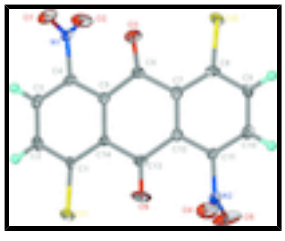


Fig. 1. Displacement ellipsoid plot (Barbour, 2001) of I at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

1,5-Dichloro-4,8-dinitroanthraquinone

Crystal data

$C_{14}H_4Cl_2N_2O_6$	F(000) = 368
M_r = 367.09	D_x = 1.824 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 2429 reflections
a = 5.9596 (6) Å	θ = 2.7–28.3°
b = 11.3897 (11) Å	μ = 0.53 mm^{-1}

c = 9.8667 (9) Å	T = 100 K
β = 93.519 (1)°	Cube, yellow
V = 668.47 (11) Å^3	0.12 × 0.12 × 0.12 mm
Z = 2	

Data collection

Bruker SMART APEX diffractometer	3028 independent reflections
Radiation source: fine-focus sealed tube	2779 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.026
ω scans	θ_{max} = 27.5°, θ_{min} = 2.1°
Absorption correction: multi-scan (*SADABS*, Sheldrick, 1996)	$h = -7 \rightarrow 7$
T_{min} = 0.940, T_{max} = 0.940	$k = -14 \rightarrow 14$
6510 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H-atom parameters constrained
$wR(F^2)$ = 0.095	$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.339P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
3028 reflections	$\Delta\rho_{max}$ = 0.54 e Å^{-3}
218 parameters	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
1 restraint	Absolute structure: Flack (Flack, 1983) parameter from 1402 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.39 (8)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.62144 (12)	0.00005 (6)	0.17540 (8)	0.02356 (19)
Cl2	0.36066 (13)	0.69418 (6)	0.31405 (8)	0.0250 (2)
O1	0.0933 (4)	0.3072 (2)	0.6211 (2)	0.0271 (5)
O2	−0.1258 (4)	0.3417 (2)	0.4440 (2)	0.0277 (5)
O3	0.2920 (4)	0.4726 (2)	0.4346 (2)	0.0339 (6)
O4	1.1628 (4)	0.3759 (2)	0.0667 (2)	0.0280 (5)
O5	0.9158 (4)	0.3677 (3)	−0.1002 (2)	0.0404 (7)
O6	0.8065 (5)	0.2199 (2)	0.1277 (3)	0.0548 (9)
N1	0.0447 (4)	0.3022 (2)	0.4997 (3)	0.0147 (5)
N2	0.9756 (4)	0.3942 (2)	0.0136 (3)	0.0163 (5)
C1	0.4613 (5)	0.0953 (3)	0.2635 (3)	0.0142 (6)
C2	0.2957 (5)	0.0447 (3)	0.3375 (3)	0.0198 (6)

H2	0.2742	−0.0379	0.3350	0.024*
C3	0.1616 (5)	0.1141 (3)	0.4149 (3)	0.0162 (7)
H3	0.0494	0.0799	0.4668	0.019*
C4	0.1953 (5)	0.2341 (3)	0.4148 (3)	0.0136 (6)
C5	0.3553 (5)	0.2890 (3)	0.3409 (3)	0.0126 (6)
C6	0.3801 (5)	0.4179 (3)	0.3468 (3)	0.0181 (6)
C7	0.5285 (4)	0.4765 (3)	0.2507 (3)	0.0150 (6)
C8	0.5362 (5)	0.5992 (3)	0.2339 (3)	0.0153 (6)
C9	0.6860 (5)	0.6510 (3)	0.1494 (3)	0.0170 (6)
H9	0.6890	0.7340	0.1399	0.020*
C10	0.8297 (5)	0.5827 (3)	0.0795 (3)	0.0183 (7)
H10	0.9342	0.6178	0.0229	0.022*
C11	0.8195 (5)	0.4621 (3)	0.0930 (3)	0.0132 (6)
C12	0.6693 (5)	0.4075 (3)	0.1766 (3)	0.0132 (6)
C13	0.6668 (5)	0.2752 (3)	0.1839 (3)	0.0224 (7)
C14	0.4913 (4)	0.2177 (3)	0.2625 (3)	0.0121 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0243 (3)	0.0116 (4)	0.0366 (4)	0.0019 (3)	0.0164 (3)	−0.0016 (3)
Cl2	0.0267 (4)	0.0132 (4)	0.0370 (4)	0.0049 (3)	0.0168 (3)	0.0022 (3)
O1	0.0307 (11)	0.0364 (13)	0.0147 (9)	0.0029 (10)	0.0041 (8)	−0.0053 (9)
O2	0.0209 (10)	0.0342 (13)	0.0276 (11)	0.0143 (10)	−0.0015 (9)	−0.0066 (10)
O3	0.0459 (13)	0.0198 (12)	0.0393 (13)	−0.0033 (10)	0.0285 (11)	−0.0054 (10)
O4	0.0191 (11)	0.0362 (14)	0.0284 (11)	0.0091 (9)	−0.0015 (9)	−0.0033 (10)
O5	0.0300 (12)	0.070 (2)	0.0210 (11)	0.0167 (12)	0.0000 (9)	−0.0185 (12)
O6	0.0656 (17)	0.0125 (12)	0.094 (2)	−0.0058 (12)	0.0693 (17)	−0.0087 (12)
N1	0.0140 (11)	0.0161 (13)	0.0147 (11)	−0.0012 (9)	0.0070 (9)	0.0004 (10)
N2	0.0167 (12)	0.0142 (13)	0.0188 (12)	−0.0046 (10)	0.0064 (10)	0.0013 (10)
C1	0.0172 (13)	0.0089 (14)	0.0168 (14)	−0.0012 (11)	0.0039 (11)	0.0026 (11)
C2	0.0225 (15)	0.0122 (15)	0.0252 (15)	−0.0012 (12)	0.0051 (12)	0.0029 (12)
C3	0.0133 (14)	0.0175 (18)	0.0183 (14)	−0.0027 (12)	0.0048 (11)	0.0054 (12)
C4	0.0109 (12)	0.0172 (16)	0.0133 (13)	0.0020 (11)	0.0036 (10)	−0.0002 (11)
C5	0.0140 (13)	0.0131 (15)	0.0106 (12)	−0.0012 (10)	0.0009 (10)	−0.0013 (10)
C6	0.0212 (14)	0.0118 (14)	0.0226 (14)	−0.0022 (11)	0.0104 (12)	−0.0021 (11)
C7	0.0144 (12)	0.0151 (17)	0.0154 (13)	−0.0035 (11)	0.0018 (10)	0.0006 (10)
C8	0.0108 (13)	0.0167 (16)	0.0186 (14)	0.0029 (11)	0.0025 (11)	−0.0036 (12)
C9	0.0208 (13)	0.0108 (14)	0.0198 (13)	−0.0027 (11)	0.0046 (11)	0.0010 (11)
C10	0.0232 (16)	0.0157 (17)	0.0166 (14)	−0.0026 (13)	0.0053 (12)	0.0008 (12)
C11	0.0136 (12)	0.0127 (15)	0.0137 (12)	−0.0025 (11)	0.0045 (10)	−0.0013 (10)
C12	0.0131 (12)	0.0129 (16)	0.0138 (13)	−0.0017 (12)	0.0013 (10)	0.0002 (10)
C13	0.0279 (15)	0.0142 (15)	0.0271 (16)	−0.0009 (12)	0.0170 (13)	−0.0024 (12)
C14	0.0109 (12)	0.0115 (16)	0.0142 (12)	0.0004 (10)	0.0039 (10)	0.0002 (10)

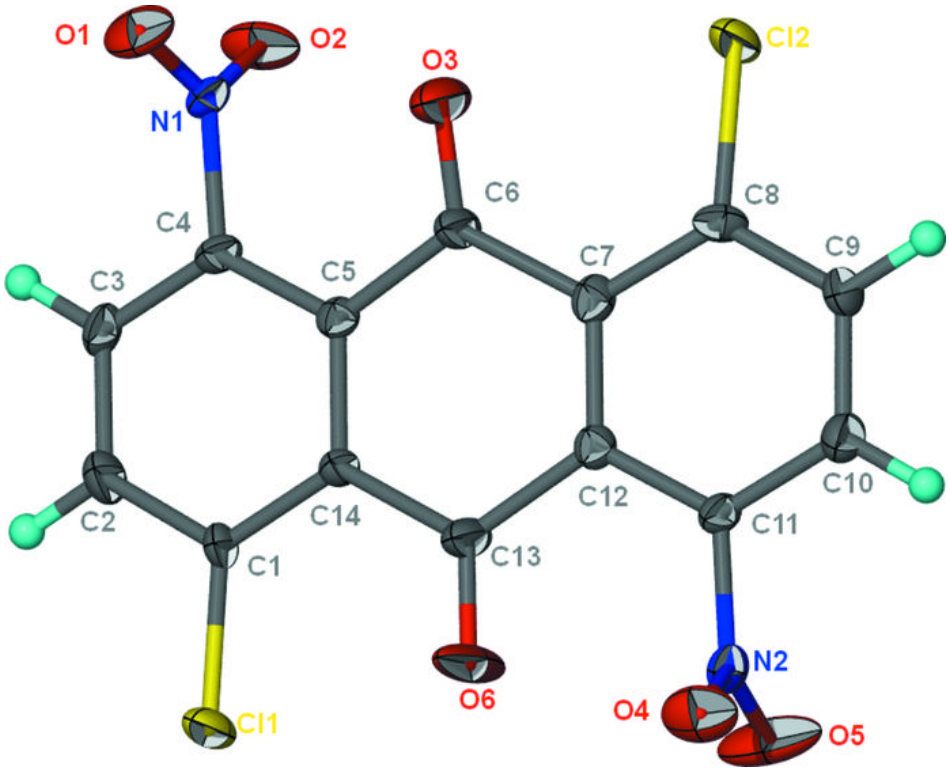
Geometric parameters (Å, °)

Cl1—C1	1.716 (3)	C3—H3	0.9500
Cl2—C8	1.730 (3)	C4—C5	1.385 (4)

O1—N1	1.216 (3)	C5—C14	1.411 (4)
O2—N1	1.211 (3)	C5—C6	1.476 (5)
O3—C6	1.213 (4)	C6—C7	1.493 (4)
O4—N2	1.221 (3)	C7—C12	1.390 (4)
O5—N2	1.197 (3)	C7—C8	1.408 (5)
O6—C13	1.205 (4)	C8—C9	1.390 (4)
N1—C4	1.483 (4)	C9—C10	1.373 (5)
N2—C11	1.472 (4)	C9—H9	0.9500
C1—C2	1.388 (4)	C10—C11	1.382 (5)
C1—C14	1.405 (5)	C10—H10	0.9500
C2—C3	1.386 (5)	C11—C12	1.399 (4)
C2—H2	0.9500	C12—C13	1.509 (5)
C3—C4	1.382 (4)	C13—C14	1.492 (4)
O2—N1—O1	124.9 (3)	C12—C7—C8	118.3 (3)
O2—N1—C4	117.3 (2)	C12—C7—C6	118.8 (3)
O1—N1—C4	117.7 (2)	C8—C7—C6	122.9 (3)
O5—N2—O4	124.8 (3)	C9—C8—C7	121.2 (3)
O5—N2—C11	118.1 (3)	C9—C8—Cl2	115.9 (3)
O4—N2—C11	116.9 (2)	C7—C8—Cl2	122.8 (3)
C2—C1—C14	120.7 (3)	C10—C9—C8	120.3 (3)
C2—C1—Cl1	116.0 (3)	C10—C9—H9	119.9
C14—C1—Cl1	123.3 (2)	C8—C9—H9	119.9
C3—C2—C1	120.4 (3)	C9—C10—C11	118.8 (3)
C3—C2—H2	119.8	C9—C10—H10	120.6
C1—C2—H2	119.8	C11—C10—H10	120.6
C4—C3—C2	118.3 (3)	C10—C11—C12	122.1 (3)
C4—C3—H3	120.8	C10—C11—N2	116.0 (3)
C2—C3—H3	120.8	C12—C11—N2	121.9 (3)
C3—C4—C5	123.4 (3)	C7—C12—C11	119.2 (3)
C3—C4—N1	115.1 (3)	C7—C12—C13	122.1 (3)
C5—C4—N1	121.5 (3)	C11—C12—C13	118.7 (3)
C4—C5—C14	117.8 (3)	O6—C13—C14	122.4 (3)
C4—C5—C6	119.8 (3)	O6—C13—C12	119.4 (3)
C14—C5—C6	122.3 (3)	C14—C13—C12	118.2 (3)
O3—C6—C5	119.6 (3)	C1—C14—C5	119.3 (3)
O3—C6—C7	121.5 (3)	C1—C14—C13	122.2 (3)
C5—C6—C7	118.7 (3)	C5—C14—C13	118.5 (3)
C14—C1—C2—C3	−2.0 (4)	C9—C10—C11—N2	179.0 (2)
Cl1—C1—C2—C3	178.3 (2)	O5—N2—C11—C10	−87.6 (4)
C1—C2—C3—C4	0.9 (4)	O4—N2—C11—C10	87.7 (4)
C2—C3—C4—C5	0.3 (5)	O5—N2—C11—C12	92.1 (4)
C2—C3—C4—N1	179.8 (2)	O4—N2—C11—C12	−92.5 (3)
O2—N1—C4—C3	−94.0 (3)	C8—C7—C12—C11	2.7 (4)
O1—N1—C4—C3	81.0 (3)	C6—C7—C12—C11	−175.8 (3)
O2—N1—C4—C5	85.4 (3)	C8—C7—C12—C13	−177.7 (3)
O1—N1—C4—C5	−99.5 (3)	C6—C7—C12—C13	3.8 (4)
C3—C4—C5—C14	−0.4 (4)	C10—C11—C12—C7	−1.3 (4)
N1—C4—C5—C14	−179.8 (2)	N2—C11—C12—C7	179.0 (2)

C3—C4—C5—C6	−179.9 (3)	C10—C11—C12—C13	179.1 (3)
N1—C4—C5—C6	0.7 (4)	N2—C11—C12—C13	−0.6 (4)
C4—C5—C6—O3	13.8 (5)	C7—C12—C13—O6	−173.0 (3)
C14—C5—C6—O3	−165.6 (3)	C11—C12—C13—O6	6.6 (5)
C4—C5—C6—C7	−170.8 (2)	C7—C12—C13—C14	6.2 (5)
C14—C5—C6—C7	9.7 (4)	C11—C12—C13—C14	−174.2 (2)
O3—C6—C7—C12	163.6 (3)	C2—C1—C14—C5	1.9 (4)
C5—C6—C7—C12	−11.6 (4)	Cl1—C1—C14—C5	−178.4 (2)
O3—C6—C7—C8	−14.9 (5)	C2—C1—C14—C13	−179.7 (3)
C5—C6—C7—C8	169.9 (3)	Cl1—C1—C14—C13	0.0 (4)
C12—C7—C8—C9	−2.3 (4)	C4—C5—C14—C1	−0.7 (4)
C6—C7—C8—C9	176.2 (3)	C6—C5—C14—C1	178.8 (3)
C12—C7—C8—Cl2	176.9 (2)	C4—C5—C14—C13	−179.1 (3)
C6—C7—C8—Cl2	−4.6 (4)	C6—C5—C14—C13	0.4 (4)
C7—C8—C9—C10	0.3 (4)	O6—C13—C14—C1	−7.4 (5)
Cl2—C8—C9—C10	−178.9 (2)	C12—C13—C14—C1	173.4 (3)
C8—C9—C10—C11	1.2 (4)	O6—C13—C14—C5	171.0 (3)
C9—C10—C11—C12	−0.7 (5)	C12—C13—C14—C5	−8.2 (4)

Fig. 1


O1
O2
N1
O3
Cl2
C6
C8
C4
C7
C9
C3
C5
C10
C12
C2
C14
C11
C1
C13
N2
O6
O4
O5
Cl1