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Aquachlorido(3,5-dinitro-2-oxidobenzoato-κ^2O^1,O^2)(1,10-phenanthroline-κ^2N,N')chromium(III)

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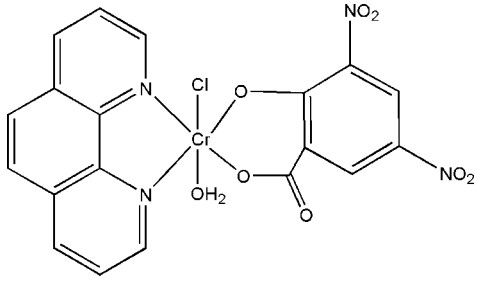
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.007 Å; R factor = 0.058; wR factor = 0.161; data-to-parameter ratio = 12.9.

In the title compound, $[Cr(C_7H_2N_2O_7)Cl(C_{12}H_8N_2)(H_2O)]$, the Cr^{III} atom displays a distorted octahedral coordination geometry, with the chelating phenantroline and 3,5-dinitrosalicylate ligands in *trans* positions. In the crystal, molecules are connected *via* O—H···O hydrogen bonds into a two-dimensional framework parallel to (100). In addition, there are π–π stacking interactions between phenanthroline ligands along the c axis, with a mean interplanar distance of 3.456 (4) Å.

Related literature

For the structure of a similar Mn^{III} complex, see: Tan & Tang (1996). For π –π stacking interactions in metal complexes, see: Janiak (2000).



Experimental

Crystal data

$[Cr(C_7H_2N_2O_7)Cl(C_{12}H_8N_2)(H_2O)]$ M_r = 511.78
Monoclinic, $P2_1/c$
a = 13.868 (7) Å
b = 16.158 (8) Å
c = 9.348 (5) Å
β = 105.947 (9)°
V = 2014.2 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.76 mm^{-1}
T = 296 K
0.19 × 0.15 × 0.13 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.869, T_{max} = 0.908
10867 measured reflections
3952 independent reflections
2378 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.161
S = 1.04
3952 reflections
306 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cr1—O3	1.906 (3)	Cr1—N1	2.056 (4)
Cr1—O1	1.926 (3)	Cr1—N2	2.065 (3)
Cr1—O8	2.017 (4)	Cr1—Cl1	2.2705 (17)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O8—H1*WB*···O6^{i}	0.83 (2)	1.94 (2)	2.759 (5)	168 (5)
O8—H1*WA*···O2^{ii}	0.81 (2)	1.81 (3)	2.581 (4)	160 (5)

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+1, -y, -z$.

Data collection: *APEX2* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2465).

References

Bruker (1997). *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2008). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3885–3896.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tan, X. S. & Tang, W. X. (1996). *Polyhedron*, **15**, 2087–2091.

supplementary materials

Acta Cryst. (2012). E**68**, m388 [doi:10.1107/S1600536812009324]

Aquachlorido(3,5-dinitro-2-oxidobenzoato-κ^2O^1,O^2)(1,10-phenanthroline-κ^2N,N')chromium(III)

Zhao-Hui Meng, Hui Lian, Shu-Shen Zhang and Yu-Quan Feng

Comment

Herein we report a mononuclear chromium(III) coordination compound $[Cr(C_{12}H_8N_2)(C_7H_2N_2O_7)Cl(H_2O)]$ (Fig. 1) obtained with the use of 3,5-dinitrosalicylic acid and 1,10-phenanthroline ligands. In the structure of title compound, the chromium atom is octahedrally coordinated by two N atoms from the phenanthroline ligand, two O atoms from the $(C_7H_2N_2O_7)^{2-}$ anion, one Cl ion and one water molecule. Bond lengths to the metal center are given in Table 1. The molecules are connected *via* O—H···O hydrogen bonds resulting in the formation of a two-dimensional supermolecular structure (Fig. 2). Moreover, there are π — π stacking interactions between phenanthroline ligands along the *c* axis due to the fact that these aromatic groups of phenanthroline ligands are parallel with each other. Such π — π stacking interactions between aromatic groups are rather popular in coordination compounds. Hydrogen bonds and π —π stacking interactions play a crucial role in stability of the crystal structure.

Experimental

All chemicals were of reagent grade quality obtained from commercial sources and used without further purification. The title compound was synthesized from a mixture of $CrCl_3.6H_2O$ (0.80 g, 3 mmol), 3,5-dinitrosalicylic acid (0.68 g, 3 mmol) and 1, 10-phenanthroline (0.60 g, 3 mmol), NaOH (0.08 g, 2 mmol) and ethanol (20 mL) by hydrothermal reaction. The mixture was stirred for half an hour, and then transferred into a Teflon-lined stainless steel autoclave (50 mL) and treated at 160 °C for 3 days. After the mixture was slowly cooled to room temperature, green block crystals suitable for X-ray structure determination were obtained.

Refinement

The H atoms bonded to C were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and with U_{iso}(H) = 1.2 times U_{eq}(C). The H atoms bonded to O atoms were located from Fourier difference maps and refined with distance restraints of O8—H1WA = 0.83 (2) Å, and O8—H1WB = 0.83 (2) Å.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

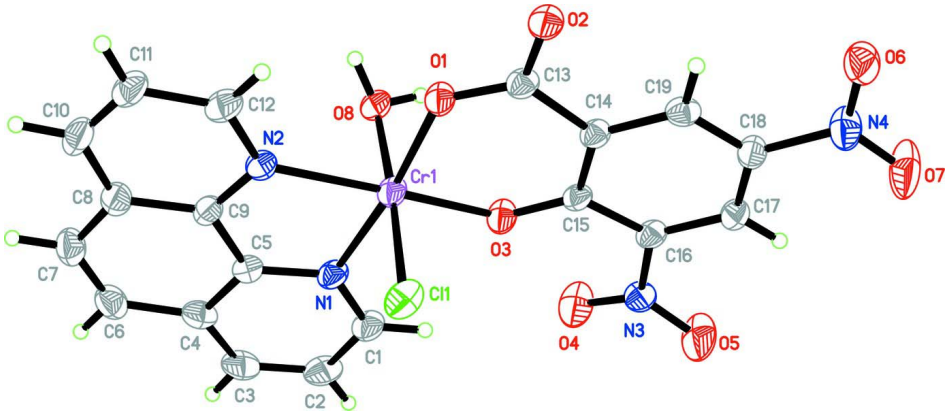


Figure 1

View of the title molecule with displacement ellipsoids drawn at the 30% probability level.

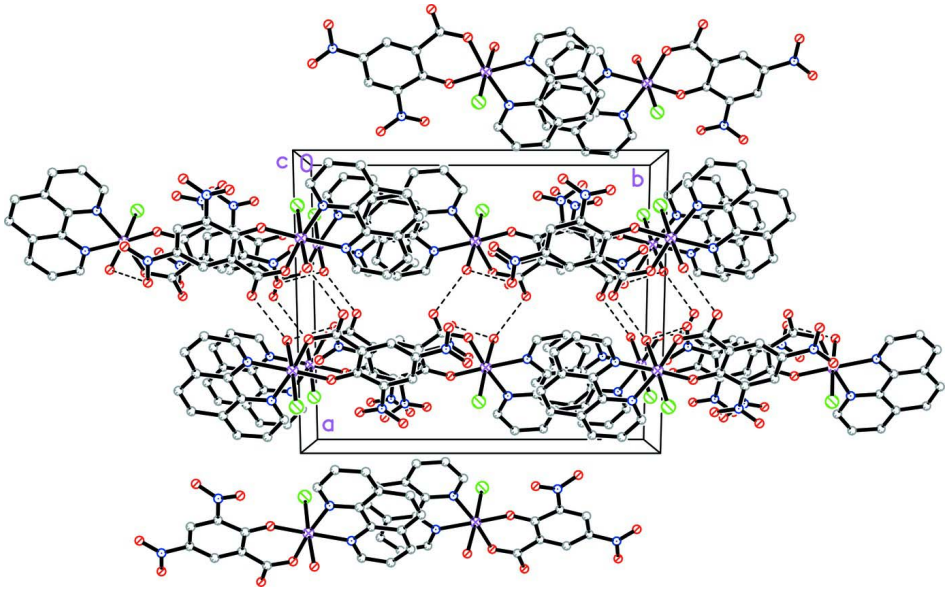


Figure 2

Crystal packing along the *c* axis. Hydrogen bonds are shown as dashed lines.

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Crystal data

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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.868$ (7) Å
$b = 16.158$ (8) Å
$c = 9.348$ (5) Å
$\beta = 105.947$ (9)°
$V = 2014.2$ (17) Å^3
$Z = 4$
$F(000) = 1036$
$D_x = 1.688$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1361 reflections
$\theta = 2.6$–20.2°
$\mu = 0.76$ mm^{-1}
$T = 296$ K
Block, green
0.19 × 0.15 × 0.13 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.869, T_{max} = 0.908
10867 measured reflections
3952 independent reflections
2378 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 26.0°, θ_{min} = 2.5°
h = −17→14
k = −19→18
l = −10→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.161
S = 1.04
3952 reflections
306 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0753P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cr1	0.71896 (5)	−0.01477 (4)	0.02803 (8)	0.0398 (2)
C1	0.8895 (3)	−0.0499 (3)	−0.1138 (5)	0.0486 (12)
H1A	0.8952	0.0071	−0.1228	0.058*
C2	0.9520 (3)	−0.1014 (3)	−0.1671 (5)	0.0549 (13)
H2A	0.9985	−0.0790	−0.2113	0.066*
C3	0.9445 (3)	−0.1854 (3)	−0.1540 (5)	0.0541 (13)
H3A	0.9861	−0.2203	−0.1892	0.065*
C4	0.8750 (3)	−0.2183 (3)	−0.0885 (5)	0.0466 (12)
C5	0.8148 (3)	−0.1623 (3)	−0.0363 (5)	0.0373 (10)
C6	0.8586 (4)	−0.3049 (3)	−0.0718 (5)	0.0538 (13)
H6A	0.8986	−0.3429	−0.1038	0.065*
C7	0.7875 (4)	−0.3331 (3)	−0.0115 (5)	0.0531 (13)
H7A	0.7769	−0.3898	−0.0076	0.064*
C8	0.7280 (4)	−0.2774 (3)	0.0467 (5)	0.0451 (12)
C9	0.7416 (3)	−0.1913 (2)	0.0324 (5)	0.0372 (10)
C10	0.6547 (4)	−0.3003 (3)	0.1147 (5)	0.0540 (14)
H10A	0.6413	−0.3562	0.1241	0.065*
C11	0.6019 (4)	−0.2423 (3)	0.1680 (5)	0.0504 (12)
H11A	0.5542	−0.2584	0.2156	0.060*
C12	0.6203 (3)	−0.1593 (3)	0.1502 (5)	0.0463 (11)

H12A	0.5840	−0.1201	0.1866	0.056*
C13	0.5958 (3)	0.1109 (3)	0.1121 (5)	0.0381 (10)
C14	0.6519 (3)	0.1771 (2)	0.0562 (4)	0.0350 (10)
C15	0.7266 (3)	0.1605 (2)	−0.0202 (5)	0.0346 (10)
C16	0.7719 (3)	0.2324 (3)	−0.0637 (5)	0.0372 (10)
C17	0.7469 (3)	0.3115 (3)	−0.0374 (5)	0.0403 (10)
H17A	0.7791	0.3564	−0.0662	0.048*
C18	0.6729 (3)	0.3229 (2)	0.0326 (5)	0.0413 (11)
C19	0.6267 (3)	0.2570 (3)	0.0788 (5)	0.0398 (10)
H19A	0.5772	0.2669	0.1265	0.048*
Cl1	0.83175 (9)	0.00500 (7)	0.25272 (14)	0.0564 (4)
N1	0.8216 (3)	−0.0795 (2)	−0.0502 (4)	0.0402 (9)
N2	0.6876 (3)	−0.1336 (2)	0.0834 (4)	0.0377 (8)
N3	0.8523 (3)	0.2233 (2)	−0.1369 (4)	0.0443 (9)
N4	0.6453 (3)	0.4060 (2)	0.0641 (5)	0.0579 (11)
O1	0.6129 (2)	0.03496 (17)	0.0950 (3)	0.0468 (8)
O2	0.5312 (2)	0.13290 (17)	0.1731 (3)	0.0468 (8)
O3	0.7505 (2)	0.08735 (17)	−0.0508 (3)	0.0462 (8)
O4	0.8663 (3)	0.1591 (2)	−0.1910 (5)	0.0756 (12)
O5	0.9030 (3)	0.2830 (2)	−0.1388 (6)	0.0961 (16)
O6	0.5745 (3)	0.4138 (2)	0.1213 (4)	0.0697 (11)
O7	0.6920 (4)	0.4640 (2)	0.0366 (5)	0.0934 (16)
O8	0.6198 (2)	−0.0388 (2)	−0.1700 (4)	0.0450 (8)
H1WB	0.608 (3)	0.0037 (19)	−0.222 (4)	0.050 (15)*
H1WA	0.565 (2)	−0.059 (3)	−0.180 (5)	0.060 (17)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.0387 (4)	0.0349 (4)	0.0546 (5)	−0.0041 (3)	0.0277 (3)	−0.0005 (3)
C1	0.040 (3)	0.050 (3)	0.063 (3)	−0.007 (2)	0.027 (2)	−0.002 (2)
C2	0.039 (3)	0.074 (4)	0.061 (3)	−0.006 (2)	0.029 (3)	−0.007 (3)
C3	0.039 (3)	0.070 (4)	0.057 (3)	0.010 (3)	0.020 (2)	−0.010 (3)
C4	0.036 (3)	0.056 (3)	0.047 (3)	0.006 (2)	0.010 (2)	−0.007 (2)
C5	0.034 (2)	0.042 (2)	0.036 (3)	−0.0025 (19)	0.0100 (19)	−0.0016 (19)
C6	0.055 (3)	0.045 (3)	0.058 (3)	0.011 (2)	0.010 (3)	−0.006 (2)
C7	0.065 (3)	0.035 (3)	0.055 (3)	0.002 (2)	0.009 (3)	−0.003 (2)
C8	0.048 (3)	0.042 (3)	0.040 (3)	−0.004 (2)	0.003 (2)	0.004 (2)
C9	0.037 (3)	0.037 (2)	0.036 (2)	−0.0067 (19)	0.007 (2)	0.0013 (19)
C10	0.057 (3)	0.046 (3)	0.051 (3)	−0.018 (3)	0.003 (3)	0.013 (2)
C11	0.049 (3)	0.056 (3)	0.047 (3)	−0.016 (2)	0.015 (2)	0.012 (2)
C12	0.042 (3)	0.057 (3)	0.044 (3)	−0.009 (2)	0.018 (2)	0.004 (2)
C13	0.035 (2)	0.042 (3)	0.041 (3)	−0.004 (2)	0.017 (2)	−0.001 (2)
C14	0.034 (2)	0.037 (2)	0.037 (3)	−0.0049 (19)	0.0147 (19)	−0.0028 (18)
C15	0.030 (2)	0.037 (2)	0.038 (3)	−0.0050 (19)	0.0117 (18)	0.0013 (18)
C16	0.035 (2)	0.041 (2)	0.040 (3)	−0.0064 (19)	0.018 (2)	−0.0002 (18)
C17	0.041 (3)	0.035 (2)	0.047 (3)	−0.005 (2)	0.015 (2)	0.0027 (19)
C18	0.045 (3)	0.034 (2)	0.047 (3)	−0.004 (2)	0.016 (2)	−0.003 (2)
C19	0.033 (2)	0.050 (3)	0.039 (3)	−0.001 (2)	0.015 (2)	−0.006 (2)
Cl1	0.0573 (8)	0.0561 (7)	0.0591 (8)	−0.0148 (6)	0.0218 (6)	−0.0049 (6)

N1	0.036 (2)	0.042 (2)	0.047 (2)	−0.0067 (16)	0.0185 (18)	−0.0006 (16)
N2	0.035 (2)	0.040 (2)	0.042 (2)	−0.0020 (16)	0.0181 (17)	0.0044 (16)
N3	0.040 (2)	0.047 (2)	0.052 (3)	−0.0023 (19)	0.0230 (19)	0.0071 (19)
N4	0.075 (3)	0.040 (2)	0.065 (3)	−0.001 (2)	0.030 (3)	−0.007 (2)
O1	0.0442 (18)	0.0342 (17)	0.076 (2)	−0.0014 (14)	0.0400 (17)	0.0005 (15)
O2	0.0453 (19)	0.0435 (17)	0.064 (2)	−0.0039 (14)	0.0364 (17)	−0.0067 (15)
O3	0.0492 (19)	0.0367 (17)	0.066 (2)	−0.0037 (14)	0.0385 (17)	−0.0027 (14)
O4	0.088 (3)	0.056 (2)	0.113 (3)	−0.011 (2)	0.078 (3)	−0.013 (2)
O5	0.090 (3)	0.052 (2)	0.182 (5)	−0.014 (2)	0.096 (3)	0.006 (3)
O6	0.070 (3)	0.050 (2)	0.103 (3)	−0.0038 (18)	0.048 (2)	−0.0191 (19)
O7	0.136 (4)	0.0358 (19)	0.144 (4)	−0.017 (2)	0.098 (3)	−0.008 (2)
O8	0.040 (2)	0.0435 (19)	0.058 (2)	−0.0070 (16)	0.0245 (17)	0.0070 (16)

Geometric parameters (Å, º)

Cr1—O3	1.906 (3)	C10—H10A	0.9300
Cr1—O1	1.926 (3)	C11—C12	1.383 (6)
Cr1—O8	2.017 (4)	C11—H11A	0.9300
Cr1—N1	2.056 (4)	C12—N2	1.325 (5)
Cr1—N2	2.065 (3)	C12—H12A	0.9300
Cr1—Cl1	2.2705 (17)	C13—O2	1.239 (5)
C1—N1	1.332 (5)	C13—O1	1.268 (5)
C1—C2	1.389 (6)	C13—C14	1.498 (6)
C1—H1A	0.9300	C14—C19	1.369 (5)
C2—C3	1.370 (7)	C14—C15	1.437 (6)
C2—H2A	0.9300	C15—O3	1.281 (5)
C3—C4	1.382 (7)	C15—C16	1.431 (5)
C3—H3A	0.9300	C16—C17	1.364 (6)
C4—C5	1.406 (6)	C16—N3	1.467 (5)
C4—C6	1.434 (6)	C17—C18	1.372 (6)
C5—N1	1.350 (5)	C17—H17A	0.9300
C5—C9	1.422 (6)	C18—C19	1.372 (6)
C6—C7	1.343 (7)	C18—N4	1.448 (5)
C6—H6A	0.9300	C19—H19A	0.9300
C7—C8	1.425 (7)	N3—O4	1.193 (4)
C7—H7A	0.9300	N3—O5	1.197 (5)
C8—C10	1.389 (7)	N4—O7	1.207 (5)
C8—C9	1.415 (6)	N4—O6	1.247 (5)
C9—N2	1.363 (5)	O8—H1WB	0.832 (19)
C10—C11	1.364 (7)	O8—H1WA	0.810 (19)
O3—Cr1—O1	92.39 (12)	C10—C11—C12	119.1 (5)
O3—Cr1—O8	89.04 (14)	C10—C11—H11A	120.4
O1—Cr1—O8	89.40 (14)	C12—C11—H11A	120.4
O3—Cr1—N1	92.74 (13)	N2—C12—C11	122.6 (5)
O1—Cr1—N1	173.41 (13)	N2—C12—H12A	118.7
O8—Cr1—N1	86.54 (14)	C11—C12—H12A	118.7
O3—Cr1—N2	171.05 (13)	O2—C13—O1	121.3 (4)
O1—Cr1—N2	94.24 (13)	O2—C13—C14	117.8 (4)
O8—Cr1—N2	85.03 (14)	O1—C13—C14	120.9 (4)

N1—Cr1—N2	80.24 (14)	C19—C14—C15	120.1 (4)
O3—Cr1—Cl1	93.60 (11)	C19—C14—C13	116.2 (4)
O1—Cr1—Cl1	91.96 (11)	C15—C14—C13	123.7 (4)
O8—Cr1—Cl1	176.97 (11)	O3—C15—C16	121.7 (4)
N1—Cr1—Cl1	91.85 (11)	O3—C15—C14	123.3 (4)
N2—Cr1—Cl1	92.18 (10)	C16—C15—C14	115.0 (4)
N1—C1—C2	122.1 (5)	C17—C16—C15	123.8 (4)
N1—C1—H1A	118.9	C17—C16—N3	116.2 (4)
C2—C1—H1A	118.9	C15—C16—N3	120.0 (4)
C3—C2—C1	119.5 (5)	C16—C17—C18	118.2 (4)
C3—C2—H2A	120.3	C16—C17—H17A	120.9
C1—C2—H2A	120.3	C18—C17—H17A	120.9
C2—C3—C4	120.0 (4)	C19—C18—C17	121.3 (4)
C2—C3—H3A	120.0	C19—C18—N4	118.9 (4)
C4—C3—H3A	120.0	C17—C18—N4	119.8 (4)
C3—C4—C5	117.3 (4)	C14—C19—C18	121.5 (4)
C3—C4—C6	125.1 (5)	C14—C19—H19A	119.2
C5—C4—C6	117.6 (4)	C18—C19—H19A	119.2
N1—C5—C4	122.7 (4)	C1—N1—C5	118.3 (4)
N1—C5—C9	116.6 (4)	C1—N1—Cr1	128.3 (3)
C4—C5—C9	120.7 (4)	C5—N1—Cr1	113.4 (3)
C7—C6—C4	122.2 (5)	C12—N2—C9	118.4 (4)
C7—C6—H6A	118.9	C12—N2—Cr1	129.4 (3)
C4—C6—H6A	118.9	C9—N2—Cr1	112.0 (3)
C6—C7—C8	121.1 (4)	O4—N3—O5	121.9 (4)
C6—C7—H7A	119.5	O4—N3—C16	121.2 (4)
C8—C7—H7A	119.5	O5—N3—C16	116.9 (4)
C10—C8—C9	116.0 (4)	O7—N4—O6	122.9 (4)
C10—C8—C7	125.4 (4)	O7—N4—C18	119.3 (4)
C9—C8—C7	118.5 (4)	O6—N4—C18	117.7 (4)
N2—C9—C8	122.7 (4)	C13—O1—Cr1	129.1 (3)
N2—C9—C5	117.5 (4)	C15—O3—Cr1	127.7 (3)
C8—C9—C5	119.8 (4)	Cr1—O8—H1WB	111 (3)
C11—C10—C8	121.2 (4)	Cr1—O8—H1WA	125 (3)
C11—C10—H10A	119.4	H1WB—O8—H1WA	103 (5)
C8—C10—H10A	119.4		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H1*WB*···O6^{i}	0.83 (2)	1.94 (2)	2.759 (5)	168 (5)
O8—H1*WA*···O2^{ii}	0.81 (2)	1.81 (3)	2.581 (4)	160 (5)

Symmetry codes: (i) x, $-y+1/2$, $z-1/2$; (ii) $-x+1$, $-y$, $-z$.