metal-organic compounds

Creatininium bis(pyridine-2,6-dicarboxylato)chromate(III) pyridine-2,6-dicarboxylic acid hexahydrate

Hossein Aghabozorg,[a] Zohreh Derikvand,[a] Marilyn M. Olmstead[b]* and Jafar Attar Gharamaleki[a]

[a]Faculty of Chemistry, Tarbiat Moallem University, Tehran, Iran, and [b]Department of Chemistry, University of California, One Shields Avenue, Davis, CA 95616-5292, USA
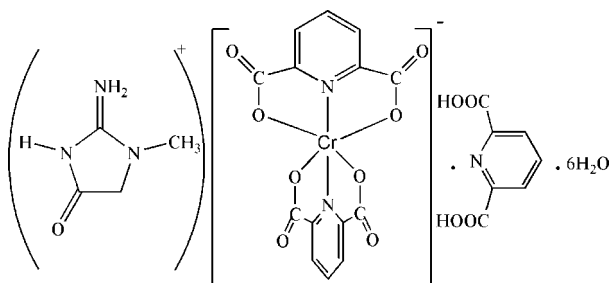
Correspondence e-mail: olmstead@chem.ucdavis.edu

The title compound, $(C_4H_8N_3O)[Cr(C_7H_3NO_4)_2] \cdot C_7H_5NO_4 \cdot 6H_2O$, was obtained by the reaction of $Cr(NO_3)_3 \cdot 9H_2O$ with pyridine-2,6-dicarboxylic acid ($pydcH_2$) and creatinine (creat) in aqueous solution (molar ratio 1:2:2). The cation is a protonated creatinine ($creatH^+$) while the anion is a bis-$pydc^{2-}$ Cr^{III} complex. The Cr^{III} is coordinated by four oxygen and two nitrogen atoms of two $(pydc)^{2-}$ groups and has a disorted octahedral coordination environment. The structure also contains a neutral molecule of $pydcH_2$ that is hydrogen bonded to the $creatH^+$ and six molecules of water. Extensive intermolecular interactions, including seventeen classical hydrogen bonds, two weak C—H⋯O bonds, and C—O⋯π stacking interactions, with O⋯centroid distances of 3.211 (13) and 3.300 (12) Å, connect the various components in the crystal structure.

Related literature

For a recent review on proton-transfer compounds and their structures, see: Aghabozorg, Manteghi et al. (2008). For related creatininium structures, see: Aghabozorg, Ramezanipour et al. (2008); Moghimi et al. (2004, 2005).

Experimental

Crystal data

$(C_4H_8N_3O)[Cr(C_7H_3NO_4)_2] \cdot$	β = 74.685 (2)°
$C_7H_5NO_4 \cdot 6H_2O$	γ = 77.644 (2)°
M_r = 771.56	V = 1589.10 (15) Å³
Triclinic, $P\bar{1}$	Z = 2
a = 9.0860 (5) Å	Mo $K\alpha$ radiation
b = 13.6274 (8) Å	μ = 0.46 mm⁻¹
c = 14.7301 (8) Å	T = 90 (2) K
α = 65.481 (2)°	0.33 × 0.32 × 0.10 mm

Data collection

Bruker SMART APEXII diffractometer	20278 measured reflections
Absorption correction: multi-scan ($SADABS$; Sheldrick, 1996)	7266 independent reflections
	6654 reflections with $I > 2\sigma(I)$
T_{min} = 0.864, T_{max} = 0.956	R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028	584 parameters
$wR(F^2)$ = 0.077	All H-atom parameters refined
S = 1.01	$\Delta\rho_{max}$ = 0.41 e Å⁻³
7266 reflections	$\Delta\rho_{min}$ = −0.46 e Å⁻³

Table 1
Selected geometric parameters (Å, °).

Cr1—N1	1.9733 (11)	Cr1—O3	1.9942 (10)
Cr1—N2	1.9769 (11)	Cr1—O1	1.9947 (9)
Cr1—O5	1.9842 (9)	Cr1—O7	1.9974 (9)
N1—Cr1—N2	172.88 (4)	N2—Cr1—O1	105.30 (4)
N1—Cr1—O5	106.16 (4)	O5—Cr1—O1	93.27 (4)
N2—Cr1—O5	79.39 (4)	O3—Cr1—O1	157.91 (4)
N1—Cr1—O3	78.84 (4)	N1—Cr1—O7	96.08 (4)
N2—Cr1—O3	96.78 (4)	N2—Cr1—O7	78.42 (4)
O5—Cr1—O3	91.39 (4)	O5—Cr1—O7	157.76 (4)
N1—Cr1—O1	79.13 (4)	O3—Cr1—O7	92.80 (4)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N4—H4A⋯O11	0.83 (2)	2.08 (2)	2.8934 (16)	167.3 (19)
N4—H4B⋯O9	0.85 (2)	1.98 (2)	2.8343 (16)	178.5 (8)
O10—H10A⋯O15	0.91 (2)	1.63 (2)	2.5382 (15)	176 (2)
O14—H14B⋯O8	0.83 (2)	2.01 (2)	2.8255 (16)	169 (2)
O15—H15A⋯O14	0.86 (2)	1.82 (2)	2.6718 (17)	168 (2)
O16—H16A⋯O4	0.78 (2)	2.07 (2)	2.8006 (15)	156 (2)
O17—H17B⋯O3	0.79 (2)	1.99 (2)	2.7758 (14)	172 (2)
O18—H18A⋯O2	0.84 (2)	1.98 (2)	2.7927 (15)	162 (2)
O19—H19A⋯O6	0.85 (3)	1.95 (3)	2.7763 (15)	164 (2)
N5—H5A⋯O16[i]	0.85 (2)	1.87 (2)	2.7175 (16)	175 (2)
O12—H12A⋯O17[ii]	0.91 (2)	1.66 (2)	2.5720 (14)	176 (2)
O14—H14A⋯O18[iii]	0.79 (3)	1.98 (3)	2.7546 (17)	167 (2)
O15—H15B⋯O7[iii]	0.77 (2)	2.16 (2)	2.9105 (15)	162 (2)
O16—H16B⋯O9[iv]	0.83 (3)	2.05 (3)	2.8083 (15)	152 (2)
O17—H17A⋯O19[v]	0.85 (2)	1.84 (2)	2.6916 (16)	178 (2)
O18—H18B⋯O8[vi]	0.84 (3)	2.12 (3)	2.9558 (15)	171 (2)
O19—H19B⋯O13[vii]	0.81 (2)	2.17 (2)	2.9531 (16)	162 (2)
C5—H5⋯O7[viii]	0.94 (2)	2.392 (19)	3.251 (2)	152.2 (15)
C10—H10⋯O6[ii]	0.929 (19)	2.283 (19)	3.0919 (17)	145.4 (18)

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y + 1, -z$; (iii) $-x, -y + 1, -z + 1$; (iv) $x, y - 1, z$; (v) $x - 1, y, z$; (vi) $x + 1, y, z$; (vii) $x + 1, y - 1, z$; (viii) $-x, -y, -z + 1$.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2533).

References

Aghabozorg, H., Manteghi, F. & Sheshmani, S. (2008). *J. Iran. Chem. Soc.* **5**, 184–227.

Aghabozorg, H., Ramezanipour, F., Soleimannejad, J., Sharif, M. A., Shokrollahi, A., Shamsipur, M., Moghimi, A., Attar Gharamaleki, J., Lippolis, V. & Blake, A. J. (2008). *Polish J. Chem.* **82**, 487–507.

Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Moghimi, A., Sharif, M. A. & Aghabozorg, H. (2004). *Acta Cryst.* E**60**, o1790–o1792.

Moghimi, A., Sharif, M. A., Shokrollahi, A., Shamsipur, M. & Aghabozorg, H. (2005). *Z. Anorg. Allg. Chem.* **631**, 902–908.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Creatininium bis(pyridine-2,6-dicarboxylato)chromate(III) pyridine-2,6-dicarboxylic acid hexahydrate

Hossein Aghabozorg, Zohreh Derikvand, Marilyn M. Olmstead and Jafar Attar Gharamaleki

S1. Comment

We have previously reported several structures that contain creatinine, pyridine-2,6-dicarboxylic acid and various metals such as: (creatH)(pydcH)·H$_2$O (Moghimi *et al.*, 2004), (creatH)$_2$[Bi(pydc)$_2$]$_2$·4H$_2$O (Moghimi *et al.*, 2005) and (creatH)[Zn(pydc)(pydcH)]· 4H$_2$O (Aghabozorg, Ramezanipour *et al.*, 2008). For more details and related literature see our recent review article (Aghabozorg, Manteghi *et al.*, 2008).

The asymmetric unit of the title compound Fig. 1, contains a [Cr(pydc)$_2$]$^-$ anion, a (creatH)$^+$ cation, a pydcH$_2$ molecule and six uncoordinated water molecules. In the anions, Cr^{III} has a N$_2$O$_4$ donor set with normal distances and angles (Table 1). The two (pydc)$^{2-}$ planes form a dihedral angle of 89.64 (1)°, and thus are virtually perpendicular to each other.

As depicted in Fig. 2, the creatininium ion is evidently strongly associated with the neutral molecule of pydcH$_2$ through two N—H⋯O hydrogen bonds and the two units are bridged by one of the water molecules. There are also two rather strong hydrogen bonds between the two carboxylic acid OH groups of the pydcH$_2$ and molecules of water. The relevant O⋯O distances are 2.5382 (15) and 2.5720 (14)Å. This structural arrangement between cation and neutral pydcH$_2$ markedly differs from the arrangement in the ion pair creatH$^+$pydcH$^-$ previously reported in the structure of the creatH$^+$pydcH$^-$ monohydrate (Moghimi *et al.*, 2004). In the latter structure, only one intramolecular hydrogen bond is formed between one of the NH$_2$ H atoms and the carboxylate group. The other NH$_2$ H atom is hydrogen bonded to the molecule of water.

In addition to numerous strong hydrogen bonds, intermolecular interactions in the the title compound include weaker C—H⋯O interactions which link the anions together, shown in Fig. 3 and Table 2, as well as C—O⋯π stacking interactions between CO groups of carboxylate fragments and aromatic rings of (pydc)$^{2-}$ with O⋯centroid distances of 3.211 (13) and 3.300 (12) Å (Fig. 4).

S2. Experimental

The reaction between pyridine-2,6-dicarboxylic acid (100 mg, 1 mmol) in 10 ml water, creatinine (creat) (110 mg, 1 mmol) in 20 ml water and chromium(III) nitrate nonahydrate (100 mg, 0.5 mmol) in 5 ml water at 2:2:1 molar ratio gave violet crystals after slow evaporation of the solvent at the room temperature.

S3. Refinement

All H atoms were freely refined with isotropic thermal parameters.
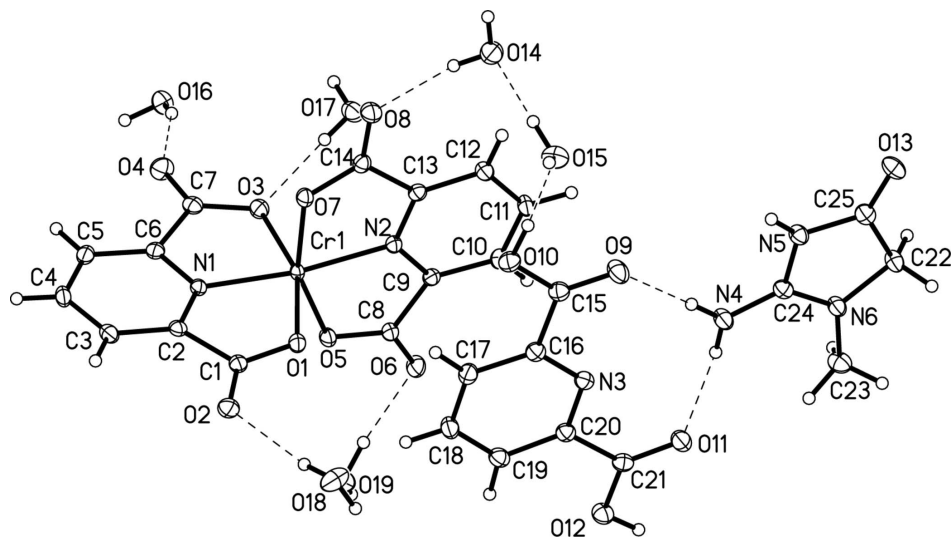
Figure 1

A view of the asymmetric unit of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.
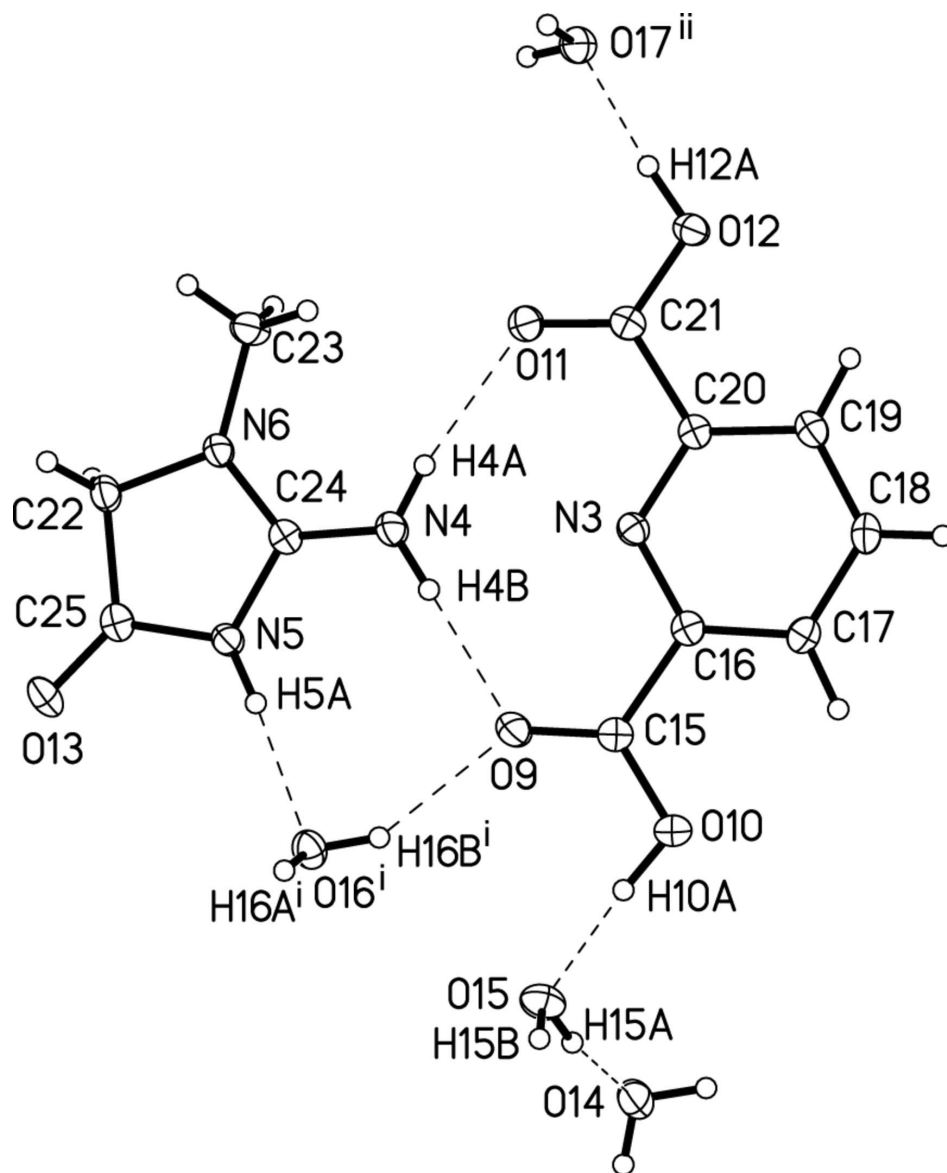
Figure 2
Details of the hydrogen bonding between the creatininium cation, the neutral pydcH$_2$ molecule and the uncoordinated water molecules. Symmetry codes: (i) x, $1 + y$, z, (ii) $1 - x$, $1 - y$, $-z$.
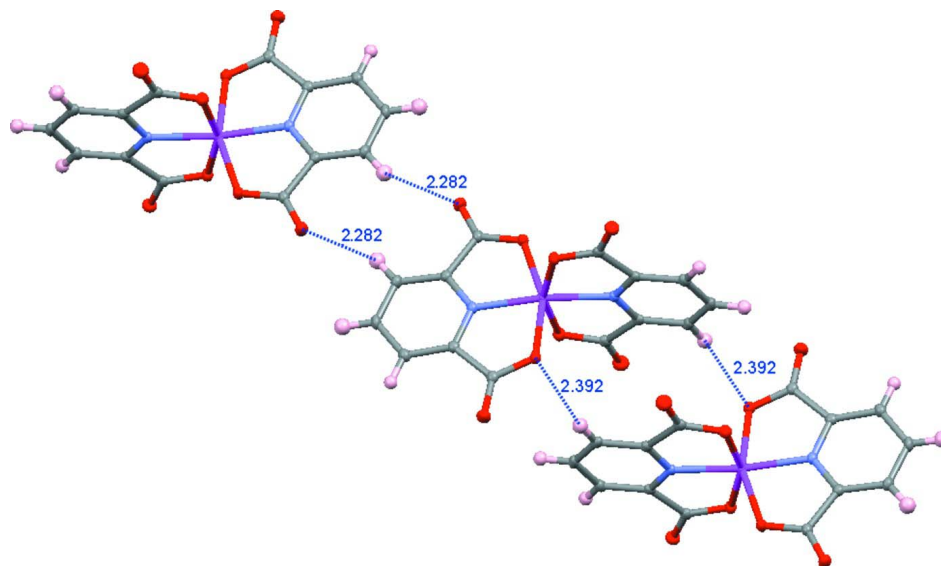
Figure 3

One dimensional chains of [Cr(pydc)$_2$]$^-$ anions that are generated by C–H···O hydrogen bonds.
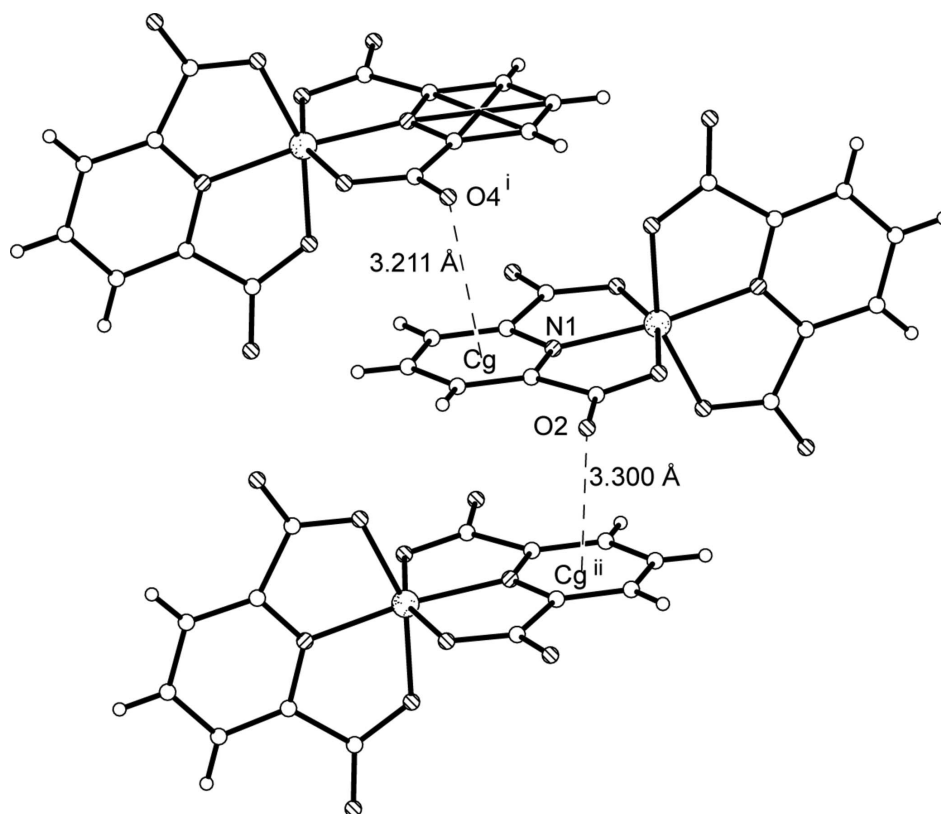
Figure 4

C–O···π stacking interactions in the title compound. Distances are reported between O atoms and the centroid (Cg) of the N1/C2-C6 aromatic ring. Symmetry codes: (i) 1-x, -y, 1-z), (ii) (-x, -y, 1-z).

Creatininium bis(pyridine-2,6-dicarboxylato)chromate(III) pyridine-2,6-dicarboxylic acid hexahydrate

Crystal data

$(C_4H_8N_3O)[Cr(C_7H_3NO_4)_2]\cdot C_7H_5NO_4\cdot 6H_2O$	$Z = 2$
$M_r = 771.56$	$F(000) = 798$
Triclinic, $P\overline{1}$	$D_x = 1.612$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.0860$ (5) Å	Cell parameters from 6379 reflections
$b = 13.6274$ (8) Å	$\theta = 3.0–31.5°$
$c = 14.7301$ (8) Å	$\mu = 0.46$ mm^{-1}
$\alpha = 65.481$ (2)°	$T = 90$ K
$\beta = 74.685$ (2)°	Plate, violet
$\gamma = 77.644$ (2)°	$0.33 \times 0.32 \times 0.10$ mm
$V = 1589.10$ (15) Å^3	

Data collection

Bruker SMART APEXII diffractometer	20278 measured reflections
Radiation source: fine-focus sealed tube	7266 independent reflections
Graphite monochromator	6654 reflections with $I > 2\sigma(I)$
Detector resolution: 8.3 pixels mm^{-1}	$R_{int} = 0.019$
ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.7°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -11\rightarrow11$
	$k = -17\rightarrow17$
$T_{min} = 0.864$, $T_{max} = 0.956$	$l = -19\rightarrow19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.028$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.077$	
$S = 1.02$	All H-atom parameters refined
7266 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0385P)^2 + 0.9325P]$ where $P = (F_o^2 + 2F_c^2)/3$
584 parameters	
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.41$ e Å^{-3}
	$\Delta\rho_{min} = -0.46$ e Å^{-3}

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}
Cr1	0.25265 (2)	0.216209 (16)	0.359634 (15)	0.01061 (6)
O1	0.36533 (10)	0.23292 (7)	0.45071 (7)	0.01405 (18)
O2	0.46030 (11)	0.14296 (8)	0.59416 (7)	0.0175 (2)

O3	0.15065 (11)	0.14097 (8)	0.30655 (7)	0.01496 (19)
O4	0.07696 (12)	−0.01935 (8)	0.33737 (8)	0.0200 (2)
O5	0.43892 (10)	0.22188 (7)	0.25150 (7)	0.01432 (19)
O6	0.55182 (11)	0.32381 (8)	0.09398 (7)	0.0181 (2)
O7	0.05282 (10)	0.26969 (7)	0.43082 (7)	0.01383 (18)
O8	−0.14469 (11)	0.40222 (8)	0.41140 (8)	0.0189 (2)
N1	0.26631 (12)	0.06706 (9)	0.46327 (8)	0.0116 (2)
N2	0.21136 (12)	0.36451 (9)	0.25887 (8)	0.0120 (2)
C1	0.38970 (14)	0.14811 (10)	0.53165 (10)	0.0130 (2)
C2	0.32378 (14)	0.04896 (10)	0.54427 (10)	0.0126 (2)
C3	0.31731 (15)	−0.05060 (11)	0.62527 (10)	0.0154 (3)
H3	0.3540 (19)	−0.0619 (13)	0.6818 (13)	0.016 (4)*
C4	0.25291 (15)	−0.13028 (11)	0.61755 (10)	0.0166 (3)
H4	0.2463 (19)	−0.1981 (14)	0.6707 (13)	0.017 (4)*
C5	0.19581 (15)	−0.11032 (11)	0.53165 (11)	0.0156 (3)
H5	0.150 (2)	−0.1614 (14)	0.5244 (13)	0.020 (4)*
C6	0.20238 (14)	−0.00750 (10)	0.45504 (10)	0.0131 (2)
C7	0.13731 (14)	0.03755 (11)	0.35899 (10)	0.0140 (2)
C8	0.44766 (15)	0.30926 (10)	0.16929 (10)	0.0133 (2)
C9	0.31394 (14)	0.39718 (10)	0.17162 (10)	0.0128 (2)
C10	0.28865 (16)	0.49990 (11)	0.09810 (10)	0.0154 (3)
H10	0.357 (2)	0.5255 (15)	0.0365 (14)	0.023 (4)*
C11	0.15425 (16)	0.56585 (11)	0.11809 (10)	0.0164 (3)
H11	0.135 (2)	0.6360 (15)	0.0687 (14)	0.023 (4)*
C12	0.04727 (15)	0.52912 (11)	0.20890 (10)	0.0152 (3)
H12	−0.0436 (19)	0.5732 (14)	0.2231 (12)	0.015 (4)*
C13	0.08062 (14)	0.42558 (10)	0.27889 (10)	0.0127 (2)
C14	−0.01527 (15)	0.36454 (10)	0.38121 (10)	0.0134 (2)
O13	0.05039 (12)	1.18680 (8)	−0.06791 (8)	0.0204 (2)
N4	0.39073 (15)	0.87378 (10)	0.02195 (10)	0.0175 (2)
H4A	0.476 (2)	0.8454 (16)	0.0006 (15)	0.029 (5)*
H4B	0.340 (2)	0.8382 (16)	0.0802 (16)	0.028 (5)*
N5	0.19415 (13)	1.02123 (9)	−0.00184 (9)	0.0156 (2)
H5A	0.140 (2)	0.9944 (16)	0.0570 (16)	0.032 (5)*
N6	0.39959 (13)	1.03426 (9)	−0.12569 (9)	0.0156 (2)
C22	0.30056 (16)	1.13631 (11)	−0.16289 (11)	0.0172 (3)
H22A	0.350 (2)	1.1955 (15)	−0.1755 (14)	0.024 (4)*
H22B	0.2690 (19)	1.1442 (13)	−0.2225 (13)	0.015 (4)*
C23	0.54951 (16)	1.00844 (13)	−0.18301 (11)	0.0193 (3)
H23A	0.540 (3)	0.9830 (19)	−0.2282 (19)	0.051 (7)*
H23B	0.617 (3)	0.959 (2)	−0.1406 (19)	0.055 (7)*
H23C	0.599 (3)	1.070 (2)	−0.217 (2)	0.065 (8)*
C24	0.33392 (15)	0.97084 (11)	−0.03381 (10)	0.0145 (2)
C25	0.16432 (16)	1.12269 (11)	−0.07475 (10)	0.0161 (3)
O9	0.22431 (12)	0.75720 (8)	0.21745 (8)	0.0222 (2)
O10	0.16856 (11)	0.60735 (8)	0.35557 (7)	0.0185 (2)
H10A	0.080 (3)	0.6511 (19)	0.3638 (17)	0.045 (6)*
O11	0.69061 (11)	0.75755 (8)	−0.01882 (8)	0.0204 (2)

O12	0.86113 (11)	0.60775 (8)	0.00786 (8)	0.0185 (2)
H12A	0.915 (3)	0.649 (2)	−0.0534 (19)	0.051 (7)*
N3	0.50100 (13)	0.65317 (9)	0.16037 (9)	0.0143 (2)
C15	0.25904 (15)	0.66364 (11)	0.27266 (10)	0.0158 (3)
C16	0.41037 (15)	0.59999 (11)	0.24827 (10)	0.0149 (3)
C17	0.44864 (16)	0.49323 (11)	0.31185 (10)	0.0167 (3)
H17	0.381 (2)	0.4595 (15)	0.3761 (14)	0.024 (4)*
C18	0.58702 (16)	0.43822 (11)	0.28135 (11)	0.0181 (3)
H18	0.613 (2)	0.3676 (16)	0.3202 (14)	0.023 (4)*
C19	0.68190 (16)	0.49118 (11)	0.18923 (11)	0.0167 (3)
H19	0.777 (2)	0.4580 (14)	0.1653 (13)	0.021 (4)*
C20	0.63432 (15)	0.59888 (11)	0.13218 (10)	0.0145 (2)
C21	0.73088 (15)	0.66371 (11)	0.03255 (10)	0.0152 (3)
O14	−0.28792 (14)	0.61850 (10)	0.36097 (10)	0.0268 (2)
H14A	−0.377 (3)	0.6268 (19)	0.3838 (18)	0.048 (7)*
H14B	−0.258 (3)	0.553 (2)	0.3801 (17)	0.041 (6)*
O15	−0.08716 (12)	0.72137 (9)	0.38192 (8)	0.0220 (2)
H15A	−0.160 (3)	0.6886 (18)	0.3832 (17)	0.040 (6)*
H15B	−0.098 (3)	0.7271 (18)	0.4335 (18)	0.039 (6)*
O16	0.00515 (12)	−0.05810 (9)	0.18249 (8)	0.0188 (2)
H16A	0.008 (3)	−0.0312 (19)	0.2190 (18)	0.041 (6)*
H16B	0.043 (3)	−0.122 (2)	0.2076 (18)	0.049 (7)*
O17	−0.02251 (12)	0.28295 (9)	0.16386 (8)	0.0189 (2)
H17A	−0.076 (3)	0.2496 (18)	0.1493 (16)	0.039 (6)*
H17B	0.029 (3)	0.2388 (19)	0.2012 (18)	0.041 (6)*
O18	0.59381 (13)	0.31757 (10)	0.57788 (9)	0.0256 (2)
H18A	0.571 (3)	0.2635 (19)	0.5733 (16)	0.040 (6)*
H18B	0.673 (3)	0.3345 (19)	0.5322 (19)	0.048 (7)*
O19	0.81371 (13)	0.17421 (9)	0.11655 (9)	0.0220 (2)
H19A	0.731 (3)	0.215 (2)	0.1020 (18)	0.050 (7)*
H19B	0.867 (3)	0.1690 (18)	0.0646 (18)	0.043 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.01120 (10)	0.00907 (10)	0.00929 (10)	−0.00114 (7)	−0.00217 (7)	−0.00125 (8)
O1	0.0153 (4)	0.0122 (4)	0.0137 (4)	−0.0024 (3)	−0.0039 (3)	−0.0029 (4)
O2	0.0175 (5)	0.0198 (5)	0.0164 (5)	−0.0028 (4)	−0.0066 (4)	−0.0059 (4)
O3	0.0158 (4)	0.0152 (4)	0.0136 (4)	−0.0018 (3)	−0.0041 (3)	−0.0045 (4)
O4	0.0214 (5)	0.0206 (5)	0.0232 (5)	−0.0033 (4)	−0.0063 (4)	−0.0118 (4)
O5	0.0134 (4)	0.0126 (4)	0.0127 (4)	−0.0005 (3)	−0.0017 (3)	−0.0016 (4)
O6	0.0162 (5)	0.0165 (5)	0.0146 (5)	−0.0017 (4)	0.0017 (4)	−0.0023 (4)
O7	0.0140 (4)	0.0125 (4)	0.0125 (4)	−0.0016 (3)	−0.0020 (3)	−0.0026 (4)
O8	0.0144 (5)	0.0185 (5)	0.0191 (5)	0.0005 (4)	−0.0003 (4)	−0.0055 (4)
N1	0.0101 (5)	0.0106 (5)	0.0120 (5)	−0.0004 (4)	−0.0012 (4)	−0.0034 (4)
N2	0.0126 (5)	0.0112 (5)	0.0116 (5)	−0.0019 (4)	−0.0032 (4)	−0.0030 (4)
C1	0.0110 (6)	0.0134 (6)	0.0124 (6)	−0.0004 (5)	−0.0004 (4)	−0.0044 (5)
C2	0.0092 (5)	0.0133 (6)	0.0131 (6)	0.0004 (4)	−0.0014 (4)	−0.0043 (5)

C3	0.0130 (6)	0.0161 (6)	0.0128 (6)	0.0011 (5)	−0.0018 (5)	−0.0029 (5)
C4	0.0145 (6)	0.0107 (6)	0.0163 (6)	0.0001 (5)	0.0008 (5)	−0.0003 (5)
C5	0.0120 (6)	0.0117 (6)	0.0204 (7)	−0.0015 (5)	0.0006 (5)	−0.0057 (5)
C6	0.0097 (5)	0.0131 (6)	0.0154 (6)	−0.0002 (4)	−0.0004 (5)	−0.0061 (5)
C7	0.0109 (6)	0.0161 (6)	0.0144 (6)	0.0006 (5)	−0.0007 (5)	−0.0072 (5)
C8	0.0137 (6)	0.0120 (6)	0.0134 (6)	−0.0020 (5)	−0.0040 (5)	−0.0029 (5)
C9	0.0123 (6)	0.0132 (6)	0.0126 (6)	−0.0029 (5)	−0.0030 (5)	−0.0036 (5)
C10	0.0161 (6)	0.0142 (6)	0.0129 (6)	−0.0037 (5)	−0.0024 (5)	−0.0017 (5)
C11	0.0194 (7)	0.0105 (6)	0.0160 (6)	−0.0018 (5)	−0.0064 (5)	−0.0002 (5)
C12	0.0151 (6)	0.0132 (6)	0.0170 (6)	−0.0002 (5)	−0.0049 (5)	−0.0050 (5)
C13	0.0124 (6)	0.0134 (6)	0.0137 (6)	−0.0023 (5)	−0.0034 (5)	−0.0056 (5)
C14	0.0143 (6)	0.0134 (6)	0.0133 (6)	−0.0031 (5)	−0.0030 (5)	−0.0050 (5)
O13	0.0199 (5)	0.0185 (5)	0.0230 (5)	0.0060 (4)	−0.0071 (4)	−0.0104 (4)
N4	0.0171 (6)	0.0156 (6)	0.0160 (6)	0.0020 (5)	−0.0035 (5)	−0.0040 (5)
N5	0.0153 (5)	0.0152 (5)	0.0145 (6)	0.0010 (4)	−0.0026 (4)	−0.0054 (5)
N6	0.0154 (5)	0.0135 (5)	0.0152 (5)	0.0013 (4)	−0.0033 (4)	−0.0041 (4)
C22	0.0189 (7)	0.0134 (6)	0.0171 (6)	0.0017 (5)	−0.0046 (5)	−0.0046 (5)
C23	0.0163 (7)	0.0232 (7)	0.0163 (7)	0.0009 (5)	−0.0020 (5)	−0.0078 (6)
C24	0.0153 (6)	0.0153 (6)	0.0149 (6)	−0.0005 (5)	−0.0052 (5)	−0.0071 (5)
C25	0.0187 (6)	0.0158 (6)	0.0168 (6)	−0.0008 (5)	−0.0065 (5)	−0.0078 (5)
O9	0.0191 (5)	0.0192 (5)	0.0199 (5)	0.0038 (4)	−0.0009 (4)	−0.0042 (4)
O10	0.0149 (5)	0.0199 (5)	0.0187 (5)	−0.0026 (4)	0.0000 (4)	−0.0071 (4)
O11	0.0169 (5)	0.0182 (5)	0.0198 (5)	0.0005 (4)	−0.0032 (4)	−0.0027 (4)
O12	0.0156 (5)	0.0191 (5)	0.0175 (5)	0.0007 (4)	−0.0008 (4)	−0.0066 (4)
N3	0.0138 (5)	0.0145 (5)	0.0154 (5)	−0.0008 (4)	−0.0040 (4)	−0.0062 (4)
C15	0.0154 (6)	0.0177 (6)	0.0151 (6)	−0.0021 (5)	−0.0036 (5)	−0.0067 (5)
C16	0.0157 (6)	0.0154 (6)	0.0156 (6)	−0.0021 (5)	−0.0044 (5)	−0.0070 (5)
C17	0.0184 (6)	0.0154 (6)	0.0163 (6)	−0.0041 (5)	−0.0037 (5)	−0.0050 (5)
C18	0.0209 (7)	0.0124 (6)	0.0200 (7)	−0.0008 (5)	−0.0073 (5)	−0.0038 (5)
C19	0.0156 (6)	0.0155 (6)	0.0206 (7)	0.0006 (5)	−0.0057 (5)	−0.0084 (5)
C20	0.0148 (6)	0.0151 (6)	0.0155 (6)	−0.0017 (5)	−0.0048 (5)	−0.0066 (5)
C21	0.0139 (6)	0.0178 (6)	0.0161 (6)	−0.0014 (5)	−0.0046 (5)	−0.0077 (5)
O14	0.0180 (6)	0.0181 (6)	0.0398 (7)	0.0000 (4)	−0.0020 (5)	−0.0100 (5)
O15	0.0191 (5)	0.0293 (6)	0.0188 (5)	−0.0005 (4)	−0.0025 (4)	−0.0122 (5)
O16	0.0224 (5)	0.0170 (5)	0.0175 (5)	0.0006 (4)	−0.0048 (4)	−0.0080 (4)
O17	0.0174 (5)	0.0201 (5)	0.0166 (5)	0.0003 (4)	−0.0054 (4)	−0.0044 (4)
O18	0.0191 (5)	0.0295 (6)	0.0342 (6)	−0.0083 (4)	0.0033 (5)	−0.0204 (5)
O19	0.0187 (5)	0.0239 (5)	0.0215 (5)	0.0022 (4)	−0.0059 (4)	−0.0080 (4)

Geometric parameters (Å, º)

Cr1—N1	1.9733 (11)	N5—C25	1.3782 (17)
Cr1—N2	1.9769 (11)	N5—H5A	0.85 (2)
Cr1—O5	1.9842 (9)	N6—C24	1.3259 (17)
Cr1—O3	1.9942 (10)	N6—C23	1.4581 (18)
Cr1—O1	1.9947 (9)	N6—C22	1.4616 (17)
Cr1—O7	1.9974 (9)	C22—C25	1.5171 (19)
O1—C1	1.3009 (15)	C22—H22A	0.938 (19)

O2—C1	1.2262 (16)	C22—H22B	0.952 (17)
O3—C7	1.3106 (16)	C23—H23A	0.90 (3)
O4—C7	1.2176 (17)	C23—H23B	0.95 (3)
O5—C8	1.2975 (15)	C23—H23C	0.92 (3)
O6—C8	1.2283 (16)	O9—C15	1.2204 (17)
O7—C14	1.3039 (16)	O10—C15	1.3107 (17)
O8—C14	1.2244 (16)	O10—H10A	0.91 (2)
N1—C6	1.3329 (17)	O11—C21	1.2184 (17)
N1—C2	1.3366 (17)	O12—C21	1.3157 (16)
N2—C13	1.3365 (17)	O12—H12A	0.91 (2)
N2—C9	1.3373 (16)	N3—C20	1.3390 (17)
C1—C2	1.5160 (18)	N3—C16	1.3391 (17)
C2—C3	1.3861 (18)	C15—C16	1.5031 (18)
C3—C4	1.394 (2)	C16—C17	1.3926 (19)
C3—H3	0.920 (17)	C17—C18	1.386 (2)
C4—C5	1.395 (2)	C17—H17	0.970 (18)
C4—H4	0.932 (17)	C18—C19	1.393 (2)
C5—C6	1.3886 (18)	C18—H18	0.909 (19)
C5—H5	0.934 (18)	C19—C20	1.3942 (19)
C6—C7	1.5145 (18)	C19—H19	0.935 (18)
C8—C9	1.5172 (18)	C20—C21	1.5099 (19)
C9—C10	1.3859 (18)	O14—H14A	0.79 (3)
C10—C11	1.3949 (19)	O14—H14B	0.83 (2)
C10—H10	0.927 (19)	O15—H15A	0.86 (2)
C11—C12	1.3965 (19)	O15—H15B	0.77 (2)
C11—H11	0.944 (18)	O16—H16A	0.78 (2)
C12—C13	1.3858 (18)	O16—H16B	0.83 (3)
C12—H12	0.942 (17)	O17—H17A	0.85 (2)
C13—C14	1.5161 (18)	O17—H17B	0.79 (2)
O13—C25	1.2149 (17)	O18—H18A	0.84 (2)
N4—C24	1.3117 (18)	O18—H18B	0.84 (3)
N4—H4A	0.83 (2)	O19—H19A	0.85 (3)
N4—H4B	0.85 (2)	O19—H19B	0.81 (2)
N5—C24	1.3731 (17)		
N1—Cr1—N2	172.88 (4)	N2—C13—C14	110.86 (11)
N1—Cr1—O5	106.16 (4)	C12—C13—C14	128.84 (12)
N2—Cr1—O5	79.39 (4)	O8—C14—O7	124.76 (12)
N1—Cr1—O3	78.84 (4)	O8—C14—C13	121.81 (12)
N2—Cr1—O3	96.78 (4)	O7—C14—C13	113.42 (11)
O5—Cr1—O3	91.39 (4)	C24—N4—H4A	120.6 (14)
N1—Cr1—O1	79.13 (4)	C24—N4—H4B	120.3 (13)
N2—Cr1—O1	105.30 (4)	H4A—N4—H4B	119.1 (19)
O5—Cr1—O1	93.27 (4)	C24—N5—C25	110.51 (11)
O3—Cr1—O1	157.91 (4)	C24—N5—H5A	123.0 (14)
N1—Cr1—O7	96.08 (4)	C25—N5—H5A	126.3 (14)
N2—Cr1—O7	78.42 (4)	C24—N6—C23	125.88 (12)
O5—Cr1—O7	157.76 (4)	C24—N6—C22	110.40 (11)

O3—Cr1—O7	92.80 (4)	C23—N6—C22	123.72 (11)
O1—Cr1—O7	91.01 (4)	N6—C22—C25	102.34 (11)
C1—O1—Cr1	117.31 (8)	N6—C22—H22A	110.6 (11)
C7—O3—Cr1	117.92 (8)	C25—C22—H22A	111.4 (11)
C8—O5—Cr1	117.46 (8)	N6—C22—H22B	111.1 (10)
C14—O7—Cr1	117.96 (8)	C25—C22—H22B	110.6 (10)
C6—N1—C2	123.01 (11)	H22A—C22—H22B	110.6 (15)
C6—N1—Cr1	118.48 (9)	N6—C23—H23A	111.2 (15)
C2—N1—Cr1	118.06 (9)	N6—C23—H23B	112.8 (15)
C13—N2—C9	123.11 (11)	H23A—C23—H23B	110 (2)
C13—N2—Cr1	118.99 (9)	N6—C23—H23C	109.5 (17)
C9—N2—Cr1	117.84 (9)	H23A—C23—H23C	109 (2)
O2—C1—O1	126.04 (12)	H23B—C23—H23C	104 (2)
O2—C1—C2	120.05 (11)	N4—C24—N6	126.64 (13)
O1—C1—C2	113.91 (11)	N4—C24—N5	122.86 (13)
N1—C2—C3	120.18 (12)	N6—C24—N5	110.49 (11)
N1—C2—C1	111.06 (11)	O13—C25—N5	125.75 (13)
C3—C2—C1	128.76 (12)	O13—C25—C22	128.00 (13)
C2—C3—C4	117.72 (12)	N5—C25—C22	106.25 (11)
C2—C3—H3	119.9 (11)	C15—O10—H10A	107.6 (14)
C4—C3—H3	122.4 (10)	C21—O12—H12A	110.0 (15)
C3—C4—C5	121.20 (12)	C20—N3—C16	117.38 (11)
C3—C4—H4	120.1 (10)	O9—C15—O10	124.43 (13)
C5—C4—H4	118.7 (11)	O9—C15—C16	122.17 (12)
C6—C5—C4	117.61 (12)	O10—C15—C16	113.34 (12)
C6—C5—H5	118.9 (11)	N3—C16—C17	123.71 (12)
C4—C5—H5	123.5 (11)	N3—C16—C15	114.63 (11)
N1—C6—C5	120.24 (12)	C17—C16—C15	121.64 (12)
N1—C6—C7	111.66 (11)	C18—C17—C16	118.11 (13)
C5—C6—C7	128.04 (12)	C18—C17—H17	121.6 (11)
O4—C7—O3	125.86 (12)	C16—C17—H17	120.3 (11)
O4—C7—C6	121.13 (12)	C17—C18—C19	119.22 (13)
O3—C7—C6	112.99 (11)	C17—C18—H18	119.9 (12)
O6—C8—O5	125.49 (12)	C19—C18—H18	120.8 (12)
O6—C8—C9	120.42 (11)	C18—C19—C20	118.15 (13)
O5—C8—C9	114.09 (11)	C18—C19—H19	122.6 (11)
N2—C9—C10	119.86 (12)	C20—C19—H19	119.2 (11)
N2—C9—C8	111.14 (11)	N3—C20—C19	123.42 (12)
C10—C9—C8	129.00 (12)	N3—C20—C21	114.59 (11)
C9—C10—C11	118.03 (12)	C19—C20—C21	122.00 (12)
C9—C10—H10	122.6 (11)	O11—C21—O12	124.35 (13)
C11—C10—H10	119.4 (11)	O11—C21—C20	122.94 (12)
C10—C11—C12	121.08 (12)	O12—C21—C20	112.71 (11)
C10—C11—H11	118.9 (11)	H14A—O14—H14B	109 (2)
C12—C11—H11	120.0 (11)	H15A—O15—H15B	110 (2)
C13—C12—C11	117.60 (12)	H16A—O16—H16B	106 (2)
C13—C12—H12	120.7 (10)	H17A—O17—H17B	108 (2)
C11—C12—H12	121.7 (10)	H18A—O18—H18B	104 (2)

N2—C13—C12	120.29 (12)	H19A—O19—H19B	108 (2)
N1—Cr1—O1—C1	−2.46 (9)	C5—C6—C7—O4	−4.4 (2)
N2—Cr1—O1—C1	171.81 (9)	N1—C6—C7—O3	−2.96 (15)
O5—Cr1—O1—C1	−108.30 (9)	C5—C6—C7—O3	174.13 (12)
O3—Cr1—O1—C1	−6.46 (16)	Cr1—O5—C8—O6	−176.91 (10)
O7—Cr1—O1—C1	93.54 (9)	Cr1—O5—C8—C9	2.87 (14)
N1—Cr1—O3—C7	0.88 (9)	C13—N2—C9—C10	1.76 (19)
N2—Cr1—O3—C7	−173.43 (9)	Cr1—N2—C9—C10	179.04 (10)
O5—Cr1—O3—C7	107.08 (9)	C13—N2—C9—C8	−177.99 (11)
O1—Cr1—O3—C7	4.89 (16)	Cr1—N2—C9—C8	−0.71 (14)
O7—Cr1—O3—C7	−94.76 (9)	O6—C8—C9—N2	178.40 (12)
N1—Cr1—O5—C8	172.87 (9)	O5—C8—C9—N2	−1.39 (16)
N2—Cr1—O5—C8	−2.55 (9)	O6—C8—C9—C10	−1.3 (2)
O3—Cr1—O5—C8	94.09 (9)	O5—C8—C9—C10	178.89 (13)
O1—Cr1—O5—C8	−107.51 (9)	N2—C9—C10—C11	−0.54 (19)
O7—Cr1—O5—C8	−6.78 (16)	C8—C9—C10—C11	179.17 (12)
N1—Cr1—O7—C14	−170.15 (9)	C9—C10—C11—C12	−0.8 (2)
N2—Cr1—O7—C14	5.26 (9)	C10—C11—C12—C13	0.9 (2)
O5—Cr1—O7—C14	9.50 (16)	C9—N2—C13—C12	−1.60 (19)
O3—Cr1—O7—C14	−91.09 (9)	Cr1—N2—C13—C12	−178.85 (9)
O1—Cr1—O7—C14	110.67 (9)	C9—N2—C13—C14	177.15 (11)
O5—Cr1—N1—C6	−90.96 (10)	Cr1—N2—C13—C14	−0.10 (14)
O3—Cr1—N1—C6	−2.76 (9)	C11—C12—C13—N2	0.21 (19)
O1—Cr1—N1—C6	178.77 (10)	C11—C12—C13—C14	−178.29 (12)
O7—Cr1—N1—C6	88.91 (10)	Cr1—O7—C14—O8	172.16 (10)
O5—Cr1—N1—C2	96.49 (9)	Cr1—O7—C14—C13	−6.72 (14)
O3—Cr1—N1—C2	−175.31 (10)	N2—C13—C14—O8	−174.56 (12)
O1—Cr1—N1—C2	6.22 (9)	C12—C13—C14—O8	4.0 (2)
O7—Cr1—N1—C2	−83.64 (9)	N2—C13—C14—O7	4.35 (15)
O5—Cr1—N2—C13	179.09 (10)	C12—C13—C14—O7	−177.04 (13)
O3—Cr1—N2—C13	88.95 (10)	C24—N6—C22—C25	1.04 (14)
O1—Cr1—N2—C13	−90.40 (10)	C23—N6—C22—C25	−178.54 (12)
O7—Cr1—N2—C13	−2.54 (9)	C23—N6—C24—N4	−0.3 (2)
O5—Cr1—N2—C9	1.70 (9)	C22—N6—C24—N4	−179.89 (13)
O3—Cr1—N2—C9	−88.45 (9)	C23—N6—C24—N5	178.95 (12)
O1—Cr1—N2—C9	92.21 (9)	C22—N6—C24—N5	−0.61 (15)
O7—Cr1—N2—C9	−179.93 (10)	C25—N5—C24—N4	179.15 (12)
Cr1—O1—C1—O2	177.87 (10)	C25—N5—C24—N6	−0.15 (16)
Cr1—O1—C1—C2	−1.13 (14)	C24—N5—C25—O13	−179.32 (13)
C6—N1—C2—C3	−0.63 (19)	C24—N5—C25—C22	0.81 (15)
Cr1—N1—C2—C3	171.56 (9)	N6—C22—C25—O13	179.04 (13)
C6—N1—C2—C1	179.57 (11)	N6—C22—C25—N5	−1.09 (14)
Cr1—N1—C2—C1	−8.24 (13)	C20—N3—C16—C17	1.25 (19)
O2—C1—C2—N1	−173.12 (11)	C20—N3—C16—C15	−176.95 (11)
O1—C1—C2—N1	5.95 (15)	O9—C15—C16—N3	−1.70 (19)
O2—C1—C2—C3	7.1 (2)	O10—C15—C16—N3	175.76 (11)
O1—C1—C2—C3	−173.83 (12)	O9—C15—C16—C17	−179.95 (13)

N1—C2—C3—C4	1.51 (19)	O10—C15—C16—C17	−2.48 (18)
C1—C2—C3—C4	−178.72 (12)	N3—C16—C17—C18	−1.4 (2)
C2—C3—C4—C5	−0.50 (19)	C15—C16—C17—C18	176.71 (12)
C3—C4—C5—C6	−1.35 (19)	C16—C17—C18—C19	0.1 (2)
C2—N1—C6—C5	−1.34 (19)	C17—C18—C19—C20	1.2 (2)
Cr1—N1—C6—C5	−173.49 (9)	C16—N3—C20—C19	0.16 (19)
C2—N1—C6—C7	176.01 (11)	C16—N3—C20—C21	−179.88 (11)
Cr1—N1—C6—C7	3.86 (14)	C18—C19—C20—N3	−1.4 (2)
C4—C5—C6—N1	2.27 (19)	C18—C19—C20—C21	178.67 (12)
C4—C5—C6—C7	−174.60 (12)	N3—C20—C21—O11	−0.66 (19)
Cr1—O3—C7—O4	179.27 (10)	C19—C20—C21—O11	179.30 (13)
Cr1—O3—C7—C6	0.84 (13)	N3—C20—C21—O12	179.64 (11)
N1—C6—C7—O4	178.53 (12)	C19—C20—C21—O12	−0.40 (18)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···O11	0.83 (2)	2.08 (2)	2.8934 (16)	167.3 (19)
N4—H4B···O9	0.85 (2)	1.98 (2)	2.8343 (16)	178.5 (8)
O10—H10A···O15	0.91 (2)	1.63 (2)	2.5382 (15)	176 (2)
O14—H14B···O8	0.83 (2)	2.01 (2)	2.8255 (16)	169 (2)
O15—H15A···O14	0.86 (2)	1.82 (2)	2.6718 (17)	168 (2)
O16—H16A···O4	0.78 (2)	2.07 (2)	2.8006 (15)	156 (2)
O17—H17B···O3	0.79 (2)	1.99 (2)	2.7758 (14)	172 (2)
O18—H18A···O2	0.84 (2)	1.98 (2)	2.7927 (15)	162 (2)
O19—H19A···O6	0.85 (3)	1.95 (3)	2.7763 (15)	164 (2)
N5—H5A···O16i	0.85 (2)	1.87 (2)	2.7175 (16)	175 (2)
O12—H12A···O17ii	0.91 (2)	1.66 (3)	2.5720 (14)	176 (2)
O14—H14A···O18iii	0.79 (3)	1.98 (3)	2.7546 (17)	167 (2)
O15—H15B···O7iii	0.77 (2)	2.16 (2)	2.9105 (15)	162 (2)
O16—H16B···O9iv	0.83 (3)	2.05 (3)	2.8083 (15)	152 (2)
O17—H17A···O19v	0.85 (2)	1.84 (2)	2.6916 (16)	178 (2)
O18—H18B···O8vi	0.84 (3)	2.12 (3)	2.9558 (15)	171 (2)
O19—H19B···O13vii	0.81 (2)	2.17 (2)	2.9531 (16)	162 (2)
C5—H5···O7viii	0.94 (2)	2.392 (19)	3.251 (2)	152.2 (15)
C10—H10···O6ii	0.929 (19)	2.283 (19)	3.0919 (17)	145.4 (18)

Symmetry codes: (i) x, $y+1$, z; (ii) $-x+1$, $-y+1$, $-z$; (iii) $-x$, $-y+1$, $-z+1$; (iv) x, $y-1$, z; (v) $x-1$, y, z; (vi) $x+1$, y, z; (vii) $x+1$, $y-1$, z; (viii) $-x$, $-y$, $-z+1$.