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# 2,6-Diaminopyridinium bis(4-hydroxypyridine-2,6-dicarboxylato-$\kappa^3O^2,N,O^6$)chromate(III) dihydrate

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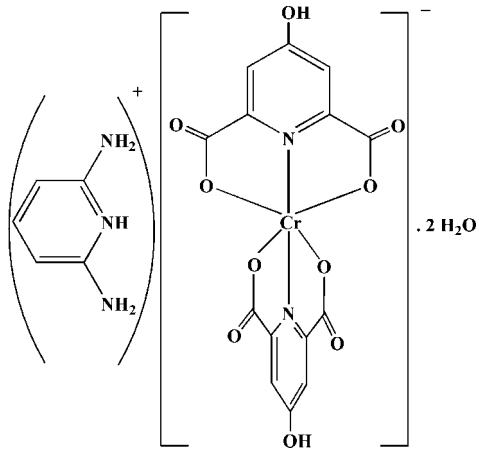
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.042; $wR$ factor = 0.105; data-to-parameter ratio = 15.6.

The reaction of chromium(III) nitrate hexahydrate, pyridine-2,6-diamine and 4-hydroxypyridine-2,6-dicarboxylic acid in a 1:2:2 molar ratio in aqueous solution resulted in the formation of the title compound, $(C_5H_8N_3)[Cr(C_7H_3NO_5)_2]\cdot 2H_2O$ or (pydaH)[Cr(hypydc)$_2$]·2H$_2$O (where pyda is pyridine-2,6-diamine and hypydcH$_2$ is 4-hydroxypyridine-2,6-dicarboxylic acid). Each Cr$^{III}$ atom is hexacoordinated by four O and two N atoms from two (hypydc)$^{2-}$ fragments, which act as tridentate ligands, in a distorted octahedral geometry. The O—Cr—O—C torsion angles between the two planes of the (hypydc)$^{2-}$ fragments [−99.81 (17) and 97.77 (17)°] indicate that these two units are almost perpendicular to one another. In the crystal structure, extensive O—H···O, N—H···O and C—H···O hydrogen bonds with $D\cdots A$ distances ranging from 2.560 (2) to 3.279 (3) Å, ion pairing, C—O···$\pi$ [O···$\pi$ = 3.166 (2) Å] and $\pi$–$\pi$ stacking interactions between (hypydc)$^{2-}$ and (pydaH)$^+$ rings [with a centroid–centroid distance of 3.3353 (14) Å] contribute to the formation of a three-dimensional supramolecular structure.

## Related literature

For related literature, see: Aghabozorg *et al.*, (2007); Aghabozorg, Manteghi *et al.* (2008); Aghabozorg, Saadaty *et al.* (2008); Ranjbar *et al.* (2001); Soleimannejad *et al.* (2008).




## Experimental

### Crystal data

$(C_5H_8N_3)[Cr(C_7H_3NO_5)_2]\cdot 2H_2O$
$M_r$ = 560.38
Monoclinic, $P2_1/n$
$a$ = 6.9590 (4) Å
$b$ = 20.9904 (12) Å
$c$ = 14.8951 (8) Å
$\beta$ = 95.3030 (13)°
$V$ = 2166.4 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.61 mm$^{-1}$
$T$ = 100 (2) K
0.32 × 0.08 × 0.07 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.829, $T_{max}$ = 0.959
24511 measured reflections
5229 independent reflections
3694 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.078

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.104
$S$ = 1.02
5229 reflections
336 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.57 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3*NA*···O2$^{i}$ | 0.87 | 2.03 | 2.840 (3) | 154 |
| N4—H4*NB*···O11$^{i}$ | 0.87 | 2.09 | 2.958 (3) | 173 |
| N4—H4*NA*···O3$^{ii}$ | 0.87 | 2.33 | 3.122 (3) | 151 |
| N5—H5*NB*···O8 | 0.87 | 1.99 | 2.831 (3) | 162 |
| N5—H5*NA*···O2$^{i}$ | 0.87 | 2.01 | 2.812 (3) | 152 |
| O5—H5*O*···O12 | 0.80 | 1.77 | 2.560 (2) | 169 |
| O10—H10*O*···O11$^{iii}$ | 0.80 | 1.83 | 2.624 (3) | 173 |
| O11—H11*C*···O1 | 0.82 | 1.92 | 2.719 (2) | 163 |
| O11—H11*D*···O7$^{iv}$ | 0.82 | 1.90 | 2.709 (3) | 169 |
| O12—H12*A*···O9$^{v}$ | 0.82 | 2.01 | 2.809 (3) | 165 |
| O12—H12*B*···O4$^{ii}$ | 0.82 | 2.05 | 2.872 (3) | 177 |
| C16—H16*A*···O3$^{ii}$ | 0.95 | 2.49 | 3.265 (3) | 139 |
| C18—H18*A*···O12$^{vi}$ | 0.95 | 2.59 | 3.279 (3) | 130 |

Symmetry codes: (i) $-x, -y+2, -z$; (ii) $x-\frac{1}{2}, -y+\frac{5}{2}, z-\frac{1}{2}$; (iii) $-x+1, -y+2, -z+1$; (iv) $x-1, y, z$; (v) $x+\frac{1}{2}, -y+\frac{5}{2}, z-\frac{1}{2}$; (vi) $x-\frac{1}{2}, -y+\frac{5}{2}, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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: YM2072).

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# supporting information

*Acta Cryst.* (2008). E**64**, m1208–m1209 [doi:10.1107/S1600536808027347]

## 2,6-Diaminopyridinium bis(4-hydroxypyridine-2,6-dicarboxylato-$\kappa^3O^2,N,O^6$)chromate(III) dihydrate

**Hossein Aghabozorg, Leila Roshan, Najmeh Firoozi, Mohammad Ghadermazi and Sara Bagheri**

### S1. Comment

The molecular structure of the title compound is shown in Fig. 1. Hydrogen bond geometries are given in Table 1. According to the crystal structure, the title compound is composed of an anionic complex, $[Cr(hypydc)_2]^-$, protonated diamino-2,6-pyridinium as a counter ion, $(pydaH)^+$, and two uncoordinated water molecules. This compound crystallizes in the monoclinic system, space group $P2_1/n$ with four molecules in the unit cell.

The $Cr^{III}$ atom is six-coordinated by two 4-hydroxypyridine-2,6-dicarboxylate, $(hypydc)^{2-}$, groups which act as tridentate ligand through one pyridine N atom and two carboxylate O atoms. N1 and N2 atoms of two $(hypydc)^{2-}$ fragments occupy the axial position, while O1, O3, O6 and O8 atoms form the equatorial plane. The N1—Cr1—N2 angle [177.33 (9)°] deviates slightly from linearity. Therefore, the coordination geometry around $Cr^{III}$ can be described as distorted octahedral. The bond angles O6—Cr1—O1, O3—Cr1—O6, O3—Cr1—O8 and O1—Cr1—O8 (93.28 (7)°, 93.82 (7)°, 90.88 (7)° and 91.46 (7)°, respectively), and the O1—Cr1—O6—C13 and O8—Cr1—O1—C6 torsion angles (-99.81 (17)° and (97.77 (17)°, respectively) show that these two $(hypydc)^{2-}$ groups are almost perpendicular. Furthermore, the bond angles O1—Cr1—O3 [156.06 (7)°] and O6—Cr1—O8 [156.99 (7)°] indicate that four carboxylate groups of the two dianions are in a flattened tetrahedral arrangement around the $Cr^{III}$ atom.

In the crystal structure of $(pydaH)[Cr(hypydc)_2].2H_2O$, the spaces between to layers of $[Cr(hypydc)_2]^-$ anions are filled with $(pydaH)^+$ cations and water molecules. There are also $\pi$-$\pi$ stacking interactions between the aromatic ring of the coordinated $(hypydc)^{2-}$ anion and the aromatic ring of the $(pydaH)^+$ cation with distances of 3.3353 (14) Å for $Cg1\cdots Cg2$. There are also C—O$\cdots\pi$ stacking interactions between C6–O2 and $Cg1$ with O$\cdots\pi$ distance of 3.166 (2) Å (1 - $x$, 2 - $y$, -$z$) [$Cg1$ and $Cg2$ are the centroid for (N1/C1—C5) and (N3/C15—C19) rings, respectively] (Fig. 2).

Intermolecular O—H···O, N—H···O and C—H···O hydrogen bonding with D···A ranging from 2.560 (2) to 3.279 (3) Å, ionpairing, $\pi$–$\pi$ and C—O$\cdots\pi$ stacking interactions seem to be effective in the stabilization of the crystal structure, resulting in the formation of an interesting supramolecular structure (Fig. 3).

### S2. Experimental

An aqueous solution of $CrCl_3$ (24 mg, 0.15 mmol), pyridine-2,6-diamine (32 mg, 0.3 mmol) and 4-Hydroxypyridine-2,6-dicarboxylic acid (54 mg, 0.3 mmol) was added to each other in a 1:2:2 molar ratio, and the reaction mixture was heated at about 50°C for 4 h. Violet crystals of the title compound were obtained from the solution after two weeks at room temperature.

### S3. Refinement

The hydrogen atoms of NH2 and OH groups and also H atoms of water molecule were found in difference Fourier synthesis. The hydrogen atoms of the H(C) atom positions were calculated. All Hydrogen atoms were refined in isotropic

approximation in riding model with the $U_{iso}$(H) parameters equal to 1.2 $U_{eq}$(Xi), where U(Xi) the equivalent thermal parameters of the carbon or nitrogen or oxygen atom to which corresponding H atom is bonded.

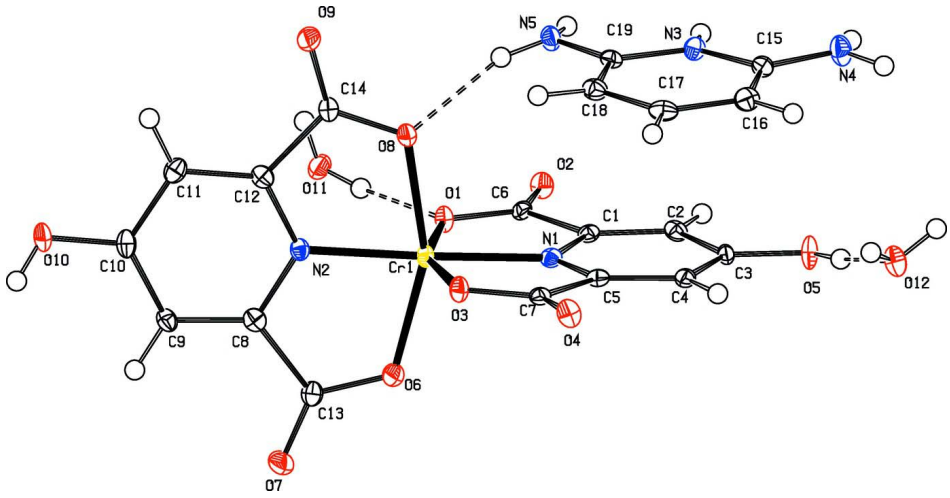


**Figure 1**

Molecular structure of (pydaH)[Cr(hypydc)$_2$].2H$_2$O, hydrogen bonds are shown as dashed lines and thermal ellipsoids are at 50% probability level.

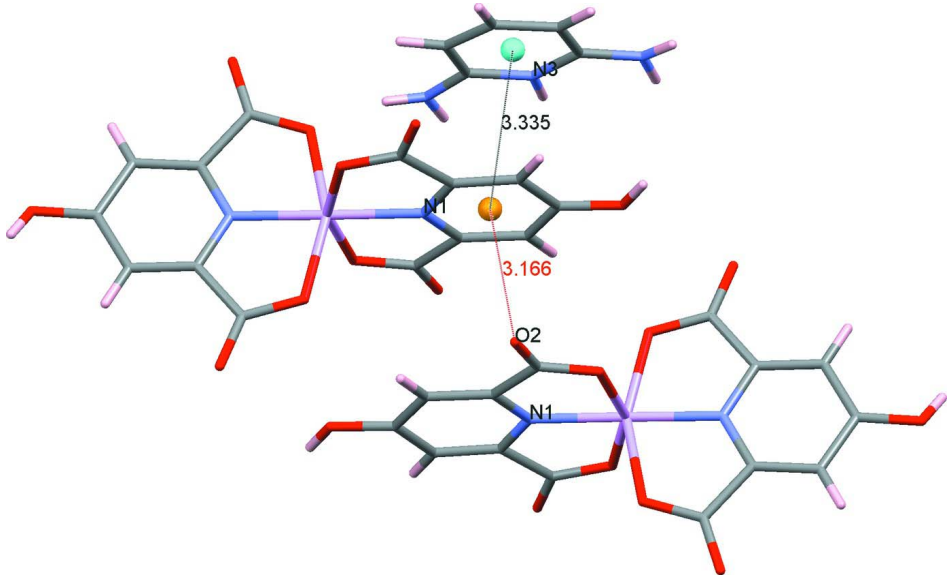


**Figure 2**

The $\pi$–$\pi$and C—O···$\pi$ stacking interactions with the distances.

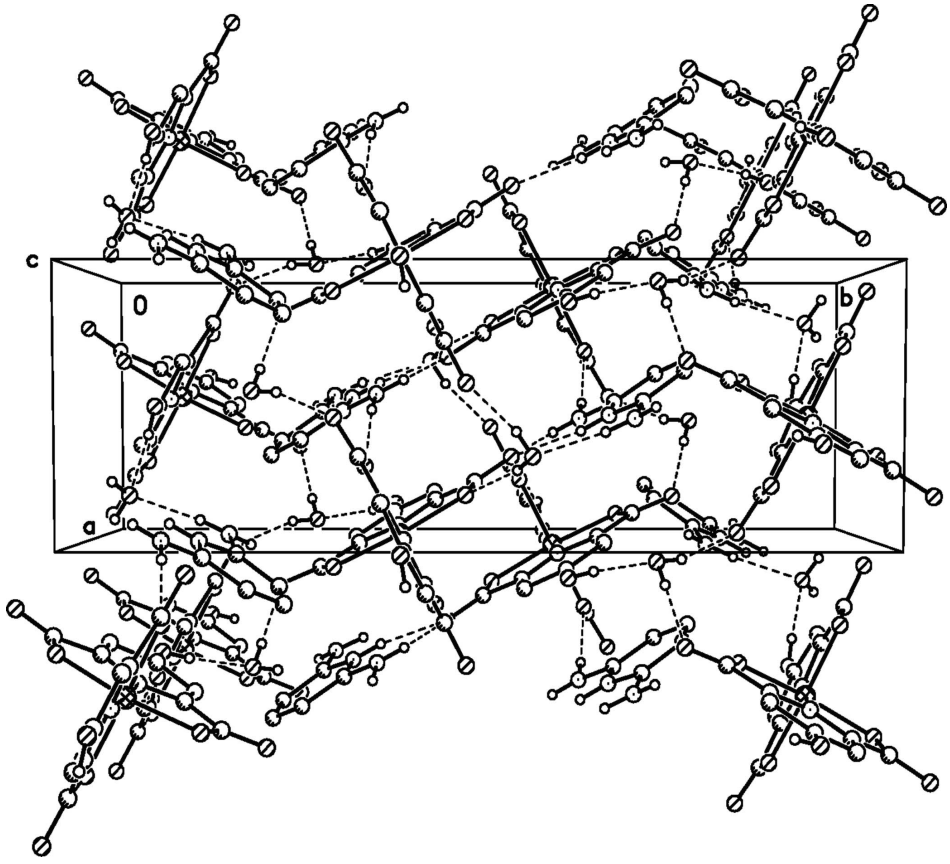


**Figure 3**

Crystal packing of the title compound showing the three dimensional H-bonded framework.

### 2,6-Diaminopyridinium bis(4-hydroxypyridine-2,6-dicarboxylato- $\kappa^3O^2,N,O^6$)chromate(III) dihydrate

*Crystal data*

$(C_5H_8N_3)[Cr(C_7H_3NO_5)_2]\cdot 2H_2O$
$M_r = 560.38$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.9590\ (4)$ Å
$b = 20.9904\ (12)$ Å
$c = 14.8951\ (8)$ Å
$\beta = 95.3030\ (13)°$
$V = 2166.4\ (2)$ Å$^3$
$Z = 4$
$F(000) = 1148$
$D_x = 1.718$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1320 reflections
$\theta = 3–28°$
$\mu = 0.61$ mm$^{-1}$
$T = 100$ K
Needle, black
0.32 × 0.08 × 0.07 mm

*Data collection*

Bruker APEXII CCD area-detector′ diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.829$, $T_{max} = 0.959$
24511 measured reflections
5229 independent reflections
3694 reflections with $I > 2\sigma(I)$
$R_{int} = 0.078$
$\theta_{max} = 28.0°$, $\theta_{min} = 1.7°$
$h = -9\rightarrow9$
$k = -27\rightarrow27$
$l = -19\rightarrow19$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.104$
$S = 1.02$
5229 reflections
336 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.9675P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.47$ e Å$^{-3}$
$\Delta\rho_{min} = -0.57$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cr1 | 0.46263 (6) | 1.098248 (19) | 0.20578 (3) | 0.00992 (10) |
| O1 | 0.3226 (2) | 1.01686 (8) | 0.17083 (11) | 0.0124 (4) |
| O2 | 0.1871 (3) | 0.95887 (8) | 0.05618 (11) | 0.0141 (4) |
| O3 | 0.5836 (2) | 1.18207 (8) | 0.18501 (11) | 0.0127 (4) |
| O4 | 0.6584 (3) | 1.25241 (8) | 0.08081 (12) | 0.0154 (4) |
| O5 | 0.3766 (3) | 1.11140 (8) | −0.20081 (11) | 0.0166 (4) |
| H5O | 0.4037 | 1.1423 | −0.2284 | 0.020* |
| O6 | 0.7149 (2) | 1.05418 (8) | 0.23449 (11) | 0.0138 (4) |
| O7 | 0.9177 (3) | 1.01550 (9) | 0.34882 (12) | 0.0192 (4) |
| O8 | 0.2157 (2) | 1.14101 (8) | 0.23077 (11) | 0.0135 (4) |
| O9 | 0.0389 (3) | 1.17639 (9) | 0.33928 (12) | 0.0174 (4) |
| O10 | 0.5003 (3) | 1.09070 (9) | 0.61131 (11) | 0.0190 (4) |
| H10O | 0.6029 | 1.0846 | 0.6388 | 0.023* |
| O11 | 0.1788 (3) | 0.93417 (8) | 0.28815 (12) | 0.0159 (4) |
| H11D | 0.0901 | 0.9544 | 0.3071 | 0.019* |
| H11C | 0.2300 | 0.9525 | 0.2480 | 0.046 (11)* |
| O12 | 0.4442 (3) | 1.21870 (8) | −0.27326 (12) | 0.0170 (4) |
| H12A | 0.4497 | 1.2508 | −0.2418 | 0.020* |
| H12B | 0.3656 | 1.2273 | −0.3160 | 0.020* |
| N1 | 0.4410 (3) | 1.10250 (10) | 0.07265 (13) | 0.0106 (4) |
| N2 | 0.4719 (3) | 1.09238 (9) | 0.33828 (13) | 0.0105 (4) |
| N3 | −0.0320 (3) | 1.16527 (10) | −0.07279 (13) | 0.0117 (4) |
| H3NA | −0.0906 | 1.1294 | −0.0859 | 0.014* |
| N4 | −0.0433 (3) | 1.19284 (11) | −0.22368 (14) | 0.0178 (5) |
| H4NB | −0.0924 | 1.1563 | −0.2413 | 0.021* |

| | | | | |
|---|---|---|---|---|
| H4NA | −0.0025 | 1.2175 | −0.2648 | 0.032 (9)* |
| N5 | −0.0454 (3) | 1.13054 (10) | 0.07407 (14) | 0.0135 (4) |
| H5NB | 0.0119 | 1.1350 | 0.1281 | 0.016* |
| H5NA | −0.0986 | 1.0961 | 0.0506 | 0.016* |
| C1 | 0.3476 (3) | 1.05598 (11) | 0.02427 (16) | 0.0101 (5) |
| C2 | 0.3233 (4) | 1.05809 (12) | −0.06822 (16) | 0.0117 (5) |
| H2A | 0.2573 | 1.0250 | −0.1019 | 0.014* |
| C3 | 0.3993 (3) | 1.11092 (11) | −0.11176 (16) | 0.0108 (5) |
| C4 | 0.4932 (3) | 1.15980 (12) | −0.05965 (16) | 0.0110 (5) |
| H4A | 0.5443 | 1.1959 | −0.0876 | 0.013* |
| C5 | 0.5088 (3) | 1.15376 (11) | 0.03232 (16) | 0.0108 (5) |
| C6 | 0.2781 (3) | 1.00555 (11) | 0.08605 (16) | 0.0105 (5) |
| C7 | 0.5931 (3) | 1.20100 (12) | 0.10225 (16) | 0.0111 (5) |
| C8 | 0.6272 (4) | 1.06604 (12) | 0.38294 (16) | 0.0123 (5) |
| C9 | 0.6455 (4) | 1.06333 (12) | 0.47589 (16) | 0.0122 (5) |
| H9A | 0.7555 | 1.0444 | 0.5078 | 0.015* |
| C10 | 0.4959 (4) | 1.08960 (11) | 0.52222 (16) | 0.0137 (5) |
| C11 | 0.3335 (4) | 1.11654 (12) | 0.47336 (17) | 0.0137 (5) |
| H11A | 0.2305 | 1.1339 | 0.5032 | 0.016* |
| C12 | 0.3287 (4) | 1.11698 (11) | 0.38087 (16) | 0.0117 (5) |
| C13 | 0.7689 (4) | 1.04231 (12) | 0.31917 (16) | 0.0128 (5) |
| C14 | 0.1782 (4) | 1.14746 (11) | 0.31450 (16) | 0.0118 (5) |
| C15 | 0.0192 (4) | 1.20629 (12) | −0.13776 (17) | 0.0132 (5) |
| C16 | 0.1308 (4) | 1.25899 (12) | −0.11129 (17) | 0.0149 (5) |
| H16A | 0.1713 | 1.2879 | −0.1549 | 0.018* |
| C17 | 0.1819 (4) | 1.26871 (12) | −0.02048 (17) | 0.0138 (5) |
| H17A | 0.2600 | 1.3044 | −0.0023 | 0.017* |
| C18 | 0.1230 (4) | 1.22815 (12) | 0.04490 (17) | 0.0135 (5) |
| H18A | 0.1551 | 1.2368 | 0.1071 | 0.016* |
| C19 | 0.0162 (4) | 1.17463 (12) | 0.01759 (16) | 0.0122 (5) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cr1 | 0.0125 (2) | 0.0104 (2) | 0.00680 (19) | 0.00029 (16) | 0.00056 (14) | −0.00067 (15) |
| O1 | 0.0170 (9) | 0.0115 (9) | 0.0083 (9) | −0.0017 (7) | 0.0000 (7) | 0.0004 (6) |
| O2 | 0.0167 (9) | 0.0123 (9) | 0.0131 (9) | −0.0027 (7) | 0.0006 (7) | −0.0010 (7) |
| O3 | 0.0161 (9) | 0.0135 (9) | 0.0084 (9) | −0.0023 (7) | 0.0003 (7) | −0.0013 (7) |
| O4 | 0.0189 (10) | 0.0120 (9) | 0.0149 (10) | −0.0027 (8) | −0.0011 (7) | 0.0010 (7) |
| O5 | 0.0284 (11) | 0.0143 (10) | 0.0068 (9) | −0.0023 (8) | 0.0011 (7) | 0.0021 (7) |
| O6 | 0.0143 (9) | 0.0156 (9) | 0.0114 (9) | 0.0019 (7) | 0.0014 (7) | −0.0015 (7) |
| O7 | 0.0170 (10) | 0.0241 (11) | 0.0161 (10) | 0.0082 (8) | −0.0007 (7) | −0.0006 (8) |
| O8 | 0.0135 (9) | 0.0175 (9) | 0.0092 (9) | 0.0023 (7) | −0.0002 (7) | −0.0007 (7) |
| O9 | 0.0177 (10) | 0.0214 (10) | 0.0134 (10) | 0.0071 (8) | 0.0029 (7) | −0.0001 (7) |
| O10 | 0.0214 (10) | 0.0279 (11) | 0.0073 (9) | 0.0029 (8) | −0.0010 (7) | −0.0002 (7) |
| O11 | 0.0169 (10) | 0.0181 (10) | 0.0136 (9) | 0.0020 (8) | 0.0052 (7) | 0.0022 (7) |
| O12 | 0.0230 (10) | 0.0145 (9) | 0.0128 (9) | −0.0013 (8) | −0.0018 (7) | 0.0020 (7) |
| N1 | 0.0102 (10) | 0.0101 (10) | 0.0117 (10) | 0.0019 (8) | 0.0014 (8) | −0.0019 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N2 | 0.0125 (10) | 0.0100 (10) | 0.0088 (10) | 0.0002 (8) | 0.0003 (8) | −0.0009 (8) |
| N3 | 0.0133 (11) | 0.0102 (10) | 0.0118 (11) | −0.0008 (8) | 0.0018 (8) | 0.0010 (8) |
| N4 | 0.0215 (12) | 0.0190 (12) | 0.0125 (11) | −0.0055 (10) | −0.0010 (9) | 0.0041 (9) |
| N5 | 0.0147 (11) | 0.0158 (11) | 0.0097 (10) | −0.0018 (9) | −0.0007 (8) | −0.0002 (8) |
| C1 | 0.0092 (12) | 0.0094 (12) | 0.0118 (12) | 0.0024 (9) | 0.0024 (9) | −0.0006 (9) |
| C2 | 0.0116 (12) | 0.0098 (12) | 0.0132 (13) | 0.0025 (9) | −0.0008 (9) | −0.0012 (9) |
| C3 | 0.0117 (12) | 0.0140 (13) | 0.0066 (11) | 0.0042 (10) | 0.0001 (9) | −0.0002 (9) |
| C4 | 0.0102 (12) | 0.0115 (12) | 0.0114 (12) | 0.0030 (9) | 0.0018 (9) | 0.0018 (9) |
| C5 | 0.0081 (12) | 0.0110 (12) | 0.0134 (13) | 0.0015 (9) | 0.0017 (9) | −0.0007 (9) |
| C6 | 0.0103 (12) | 0.0116 (12) | 0.0099 (12) | 0.0025 (10) | 0.0030 (9) | 0.0002 (9) |
| C7 | 0.0088 (12) | 0.0133 (12) | 0.0113 (12) | 0.0031 (10) | 0.0011 (9) | −0.0018 (9) |
| C8 | 0.0129 (13) | 0.0105 (12) | 0.0134 (13) | −0.0018 (10) | −0.0002 (10) | −0.0004 (9) |
| C9 | 0.0140 (13) | 0.0112 (12) | 0.0110 (12) | 0.0022 (10) | −0.0014 (9) | 0.0005 (9) |
| C10 | 0.0201 (13) | 0.0110 (12) | 0.0098 (12) | −0.0014 (10) | 0.0008 (10) | 0.0015 (9) |
| C11 | 0.0160 (13) | 0.0123 (12) | 0.0132 (13) | −0.0016 (10) | 0.0037 (10) | −0.0013 (9) |
| C12 | 0.0122 (12) | 0.0100 (12) | 0.0124 (12) | −0.0020 (9) | −0.0011 (9) | −0.0021 (9) |
| C13 | 0.0154 (13) | 0.0117 (12) | 0.0113 (12) | −0.0009 (10) | 0.0011 (10) | −0.0017 (9) |
| C14 | 0.0145 (13) | 0.0109 (12) | 0.0098 (12) | −0.0008 (10) | 0.0005 (9) | 0.0003 (9) |
| C15 | 0.0130 (13) | 0.0143 (13) | 0.0123 (13) | 0.0050 (10) | 0.0016 (10) | 0.0030 (9) |
| C16 | 0.0150 (13) | 0.0125 (13) | 0.0173 (14) | 0.0019 (10) | 0.0030 (10) | 0.0039 (10) |
| C17 | 0.0111 (12) | 0.0088 (12) | 0.0214 (14) | 0.0015 (10) | 0.0010 (10) | −0.0017 (10) |
| C18 | 0.0120 (13) | 0.0130 (13) | 0.0154 (13) | 0.0014 (10) | 0.0000 (10) | −0.0025 (10) |
| C19 | 0.0090 (12) | 0.0152 (13) | 0.0123 (13) | 0.0034 (10) | −0.0001 (9) | −0.0002 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cr1—N2 | 1.973 (2) | N4—C15 | 1.343 (3) |
| Cr1—N1 | 1.977 (2) | N4—H4NB | 0.8700 |
| Cr1—O3 | 1.9872 (17) | N4—H4NA | 0.8700 |
| Cr1—O6 | 1.9957 (18) | N5—C19 | 1.347 (3) |
| Cr1—O8 | 2.0036 (17) | N5—H5NB | 0.8700 |
| Cr1—O1 | 2.0111 (17) | N5—H5NA | 0.8701 |
| O1—C6 | 1.294 (3) | C1—C2 | 1.373 (3) |
| O2—C6 | 1.227 (3) | C1—C6 | 1.511 (3) |
| O3—C7 | 1.303 (3) | C2—C3 | 1.412 (3) |
| O4—C7 | 1.225 (3) | C2—H2A | 0.9500 |
| O5—C3 | 1.321 (3) | C3—C4 | 1.410 (3) |
| O5—H5O | 0.7999 | C4—C5 | 1.370 (3) |
| O6—C13 | 1.306 (3) | C4—H4A | 0.9500 |
| O7—C13 | 1.225 (3) | C5—C7 | 1.516 (3) |
| O8—C14 | 1.305 (3) | C8—C9 | 1.380 (3) |
| O9—C14 | 1.229 (3) | C8—C13 | 1.515 (3) |
| O10—C10 | 1.325 (3) | C9—C10 | 1.413 (3) |
| O10—H10O | 0.8000 | C9—H9A | 0.9500 |
| O11—H11D | 0.8199 | C10—C11 | 1.405 (4) |
| O11—H11C | 0.8201 | C11—C12 | 1.375 (3) |
| O12—H12A | 0.8198 | C11—H11A | 0.9500 |
| O12—H12B | 0.8201 | C12—C14 | 1.514 (3) |

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| N1—C5 | 1.339 (3) | C15—C16 | 1.388 (4) |
| N1—C1 | 1.345 (3) | C16—C17 | 1.382 (4) |
| N2—C12 | 1.334 (3) | C16—H16A | 0.9500 |
| N2—C8 | 1.335 (3) | C17—C18 | 1.384 (4) |
| N3—C15 | 1.367 (3) | C17—H17A | 0.9500 |
| N3—C19 | 1.371 (3) | C18—C19 | 1.387 (3) |
| N3—H3NA | 0.8701 | C18—H18A | 0.9500 |

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|---|---|---|---|
| N2—Cr1—N1 | 177.33 (9) | C5—C4—H4A | 120.9 |
| N2—Cr1—O3 | 103.64 (8) | C3—C4—H4A | 120.9 |
| N1—Cr1—O3 | 78.35 (8) | N1—C5—C4 | 121.6 (2) |
| N2—Cr1—O6 | 79.04 (8) | N1—C5—C7 | 110.3 (2) |
| N1—Cr1—O6 | 102.70 (8) | C4—C5—C7 | 128.0 (2) |
| O3—Cr1—O6 | 93.82 (7) | O2—C6—O1 | 124.7 (2) |
| N2—Cr1—O8 | 77.96 (8) | O2—C6—C1 | 121.4 (2) |
| N1—Cr1—O8 | 100.31 (8) | O1—C6—C1 | 113.9 (2) |
| O3—Cr1—O8 | 90.88 (7) | O4—C7—O3 | 124.6 (2) |
| O6—Cr1—O8 | 156.99 (7) | O4—C7—C5 | 121.8 (2) |
| N2—Cr1—O1 | 100.15 (8) | O3—C7—C5 | 113.6 (2) |
| N1—Cr1—O1 | 77.79 (8) | N2—C8—C9 | 120.8 (2) |
| O3—Cr1—O1 | 156.06 (7) | N2—C8—C13 | 111.6 (2) |
| O6—Cr1—O1 | 93.28 (7) | C9—C8—C13 | 127.6 (2) |
| O8—Cr1—O1 | 91.46 (7) | C8—C9—C10 | 118.0 (2) |
| C6—O1—Cr1 | 118.37 (15) | C8—C9—H9A | 121.0 |
| C7—O3—Cr1 | 118.46 (15) | C10—C9—H9A | 121.0 |
| C3—O5—H5O | 120.7 | O10—C10—C11 | 117.1 (2) |
| C13—O6—Cr1 | 117.65 (15) | O10—C10—C9 | 123.1 (2) |
| C14—O8—Cr1 | 118.36 (15) | C11—C10—C9 | 119.8 (2) |
| C10—O10—H10O | 116.5 | C12—C11—C10 | 117.7 (2) |
| H11D—O11—H11C | 113.6 | C12—C11—H11A | 121.1 |
| H12A—O12—H12B | 104.7 | C10—C11—H11A | 121.1 |
| C5—N1—C1 | 121.1 (2) | N2—C12—C11 | 121.6 (2) |
| C5—N1—Cr1 | 119.27 (16) | N2—C12—C14 | 110.8 (2) |
| C1—N1—Cr1 | 119.54 (16) | C11—C12—C14 | 127.5 (2) |
| C12—N2—C8 | 121.9 (2) | O7—C13—O6 | 126.4 (2) |
| C12—N2—Cr1 | 119.67 (17) | O7—C13—C8 | 120.2 (2) |
| C8—N2—Cr1 | 118.33 (16) | O6—C13—C8 | 113.4 (2) |
| C15—N3—C19 | 123.3 (2) | O9—C14—O8 | 124.9 (2) |
| C15—N3—H3NA | 122.3 | O9—C14—C12 | 122.0 (2) |
| C19—N3—H3NA | 114.3 | O8—C14—C12 | 113.1 (2) |
| C15—N4—H4NB | 123.8 | N4—C15—N3 | 117.4 (2) |
| C15—N4—H4NA | 116.6 | N4—C15—C16 | 124.2 (2) |
| H4NB—N4—H4NA | 117.1 | N3—C15—C16 | 118.5 (2) |
| C19—N5—H5NB | 111.0 | C17—C16—C15 | 118.9 (2) |
| C19—N5—H5NA | 117.8 | C17—C16—H16A | 120.6 |
| H5NB—N5—H5NA | 127.1 | C15—C16—H16A | 120.6 |
| N1—C1—C2 | 121.5 (2) | C16—C17—C18 | 122.1 (2) |
| N1—C1—C6 | 110.4 (2) | C16—C17—H17A | 118.9 |

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| C2—C1—C6 | 128.1 (2) | C18—C17—H17A | 118.9 |
| C1—C2—C3 | 118.0 (2) | C17—C18—C19 | 118.5 (2) |
| C1—C2—H2A | 121.0 | C17—C18—H18A | 120.7 |
| C3—C2—H2A | 121.0 | C19—C18—H18A | 120.7 |
| O5—C3—C4 | 123.6 (2) | N5—C19—N3 | 116.8 (2) |
| O5—C3—C2 | 116.9 (2) | N5—C19—C18 | 124.5 (2) |
| C4—C3—C2 | 119.5 (2) | N3—C19—C18 | 118.7 (2) |
| C5—C4—C3 | 118.2 (2) | | |

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| N2—Cr1—O1—C6 | 175.80 (17) | C3—C4—C5—C7 | −176.5 (2) |
| N1—Cr1—O1—C6 | −2.46 (16) | Cr1—O1—C6—O2 | −177.36 (19) |
| O3—Cr1—O1—C6 | 2.3 (3) | Cr1—O1—C6—C1 | 2.3 (3) |
| O6—Cr1—O1—C6 | −104.75 (17) | N1—C1—C6—O2 | 179.2 (2) |
| O8—Cr1—O1—C6 | 97.77 (17) | C2—C1—C6—O2 | −0.1 (4) |
| N2—Cr1—O3—C7 | −177.73 (16) | N1—C1—C6—O1 | −0.5 (3) |
| N1—Cr1—O3—C7 | 0.44 (17) | C2—C1—C6—O1 | −179.8 (2) |
| O6—Cr1—O3—C7 | 102.64 (17) | Cr1—O3—C7—O4 | 177.52 (19) |
| O8—Cr1—O3—C7 | −99.91 (17) | Cr1—O3—C7—C5 | −0.6 (3) |
| O1—Cr1—O3—C7 | −4.3 (3) | N1—C5—C7—O4 | −177.7 (2) |
| N2—Cr1—O6—C13 | −0.10 (17) | C4—C5—C7—O4 | 0.4 (4) |
| N1—Cr1—O6—C13 | −178.02 (17) | N1—C5—C7—O3 | 0.5 (3) |
| O3—Cr1—O6—C13 | 103.07 (17) | C4—C5—C7—O3 | 178.6 (2) |
| O8—Cr1—O6—C13 | 1.7 (3) | C12—N2—C8—C9 | −0.1 (4) |
| O1—Cr1—O6—C13 | −99.81 (17) | Cr1—N2—C8—C9 | 176.98 (18) |
| N2—Cr1—O8—C14 | 2.31 (17) | C12—N2—C8—C13 | −179.5 (2) |
| N1—Cr1—O8—C14 | −179.77 (17) | Cr1—N2—C8—C13 | −2.4 (3) |
| O3—Cr1—O8—C14 | −101.46 (17) | N2—C8—C9—C10 | −0.4 (4) |
| O6—Cr1—O8—C14 | 0.5 (3) | C13—C8—C9—C10 | 178.9 (2) |
| O1—Cr1—O8—C14 | 102.37 (17) | C8—C9—C10—O10 | −178.7 (2) |
| O3—Cr1—N1—C5 | −0.11 (17) | C8—C9—C10—C11 | 0.9 (4) |
| O6—Cr1—N1—C5 | −91.45 (18) | O10—C10—C11—C12 | 178.6 (2) |
| O8—Cr1—N1—C5 | 88.65 (18) | C9—C10—C11—C12 | −1.0 (4) |
| O1—Cr1—N1—C5 | 177.91 (19) | C8—N2—C12—C11 | 0.0 (4) |
| O3—Cr1—N1—C1 | −175.84 (19) | Cr1—N2—C12—C11 | −176.99 (18) |
| O6—Cr1—N1—C1 | 92.83 (18) | C8—N2—C12—C14 | 176.2 (2) |
| O8—Cr1—N1—C1 | −87.08 (18) | Cr1—N2—C12—C14 | −0.8 (3) |
| O1—Cr1—N1—C1 | 2.18 (17) | C10—C11—C12—N2 | 0.5 (4) |
| O3—Cr1—N2—C12 | 87.31 (19) | C10—C11—C12—C14 | −175.0 (2) |
| O6—Cr1—N2—C12 | 178.62 (19) | Cr1—O6—C13—O7 | 179.5 (2) |
| O8—Cr1—N2—C12 | −0.64 (18) | Cr1—O6—C13—C8 | −1.1 (3) |
| O1—Cr1—N2—C12 | −89.97 (19) | N2—C8—C13—O7 | −178.4 (2) |
| O3—Cr1—N2—C8 | −89.81 (18) | C9—C8—C13—O7 | 2.3 (4) |
| O6—Cr1—N2—C8 | 1.50 (18) | N2—C8—C13—O6 | 2.2 (3) |
| O8—Cr1—N2—C8 | −177.77 (19) | C9—C8—C13—O6 | −177.1 (2) |
| O1—Cr1—N2—C8 | 92.91 (18) | Cr1—O8—C14—O9 | 174.9 (2) |
| C5—N1—C1—C2 | 2.1 (3) | Cr1—O8—C14—C12 | −3.3 (3) |
| Cr1—N1—C1—C2 | 177.73 (18) | N2—C12—C14—O9 | −175.7 (2) |
| C5—N1—C1—C6 | −177.3 (2) | C11—C12—C14—O9 | 0.2 (4) |

| | | | |
|---|---|---|---|
| Cr1—N1—C1—C6 | −1.6 (3) | N2—C12—C14—O8 | 2.6 (3) |
| N1—C1—C2—C3 | −0.1 (3) | C11—C12—C14—O8 | 178.5 (2) |
| C6—C1—C2—C3 | 179.1 (2) | C19—N3—C15—N4 | −177.6 (2) |
| C1—C2—C3—O5 | 179.0 (2) | C19—N3—C15—C16 | 2.1 (4) |
| C1—C2—C3—C4 | −1.2 (3) | N4—C15—C16—C17 | 178.2 (2) |
| O5—C3—C4—C5 | −179.6 (2) | N3—C15—C16—C17 | −1.5 (4) |
| C2—C3—C4—C5 | 0.6 (3) | C15—C16—C17—C18 | −1.0 (4) |
| C1—N1—C5—C4 | −2.7 (3) | C16—C17—C18—C19 | 2.8 (4) |
| Cr1—N1—C5—C4 | −178.40 (18) | C15—N3—C19—N5 | 179.4 (2) |
| C1—N1—C5—C7 | 175.5 (2) | C15—N3—C19—C18 | −0.3 (4) |
| Cr1—N1—C5—C7 | −0.2 (3) | C17—C18—C19—N5 | 178.2 (2) |
| C3—C4—C5—N1 | 1.4 (4) | C17—C18—C19—N3 | −2.1 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3*NA*···O2$^{i}$ | 0.87 | 2.03 | 2.840 (3) | 154 |
| N4—H4*NB*···O11$^{i}$ | 0.87 | 2.09 | 2.958 (3) | 173 |
| N4—H4*NA*···O3$^{ii}$ | 0.87 | 2.33 | 3.122 (3) | 151 |
| N5—H5*NB*···O8 | 0.87 | 1.99 | 2.831 (3) | 162 |
| N5—H5*NA*···O2$^{i}$ | 0.87 | 2.01 | 2.812 (3) | 152 |
| O5—H5*O*···O12 | 0.80 | 1.77 | 2.560 (2) | 169 |
| O10—H10*O*···O11$^{iii}$ | 0.80 | 1.83 | 2.624 (3) | 173 |
| O11—H11*C*···O1 | 0.82 | 1.92 | 2.719 (2) | 163 |
| O11—H11*D*···O7$^{iv}$ | 0.82 | 1.90 | 2.709 (3) | 169 |
| O12—H12*A*···O9$^{v}$ | 0.82 | 2.01 | 2.809 (3) | 165 |
| O12—H12*B*···O4$^{ii}$ | 0.82 | 2.05 | 2.872 (3) | 177 |
| C16—H16*A*···O3$^{ii}$ | 0.95 | 2.49 | 3.265 (3) | 139 |
| C18—H18*A*···O12$^{vi}$ | 0.95 | 2.59 | 3.279 (3) | 130 |

Symmetry codes: (i) −*x*, −*y*+2, −*z*; (ii) *x*−1/2, −*y*+5/2, *z*−1/2; (iii) −*x*+1, −*y*+2, −*z*+1; (iv) *x*−1, *y*, *z*; (v) *x*+1/2, −*y*+5/2, *z*−1/2; (vi) *x*−1/2, −*y*+5/2, *z*+1/2.