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2-(2-Hydroxy-1-naphthyldiazenyl)-pyridinium [1-(2-pyridyldiazenyl)-2-naphtholato-κ^3N,N',O]tris(thiocyanato-κN)chromium(III) methanol solvate

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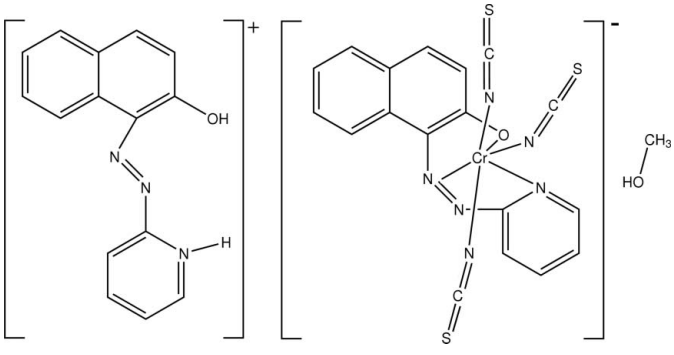
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.012 Å; R factor = 0.067; wR factor = 0.209; data-to-parameter ratio = 17.1.

In the title compound, $(C_{15}H_{12}N_3O)[Cr(C_{15}H_{10}N_3O)(NCS)_3]\cdot CH_4O$, the Cr^{III} atom is coordinated by two N atoms and one O atom from the tridentate 1-(2-pyridyldiazenyl)-2-naphtholate (PAN) ligand and three N atoms from thiocyanate ions in a distorted octahedral arrangement. Another uncoordinated and protonated PAN molecule and a methanol solvent molecule are present in the asymmetric unit. The crystal packing is stabilized by π–π interactions [centroid–centroid separation 3.594 (4) Å] and O—H···N and O—H···S hydrogen bonds.



Experimental

Crystal data

$(C_{15}H_{12}N_3O)[Cr(C_{15}H_{10}N_3O)(NCS)_3]\cdot CH_4O$
M_r = 756.82
Triclinic, $P\overline{1}$
a = 11.5045 (11) Å
b = 12.3189 (11) Å
c = 12.5197 (12) Å
α = 95.568 (2)°
β = 91.851 (2)°
γ = 96.973 (2)°
V = 1751.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.56 mm^{-1}
T = 273 (2) K
0.25 × 0.22 × 0.17 mm

Data collection

Bruker SMART APEX II CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.874, T_{max} = 0.912
11793 measured reflections
7770 independent reflections
2997 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.209
S = 0.96
7770 reflections
454 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr1—O1	1.964 (3)	Cr1—N1	2.003 (4)
Cr1—N4	1.981 (5)	Cr1—N6	2.003 (5)
Cr1—N5	1.992 (4)	Cr1—N3	2.058 (4)
O1—Cr1—N4	92.38 (16)	N5—Cr1—N6	91.81 (17)
O1—Cr1—N5	100.34 (15)	N1—Cr1—N6	91.51 (16)
N4—Cr1—N5	89.61 (17)	O1—Cr1—N3	157.01 (15)
O1—Cr1—N1	80.57 (14)	N4—Cr1—N3	89.21 (17)
N4—Cr1—N1	87.03 (16)	N5—Cr1—N3	102.61 (16)
N5—Cr1—N1	176.56 (16)	N1—Cr1—N3	76.61 (15)
O1—Cr1—N6	89.83 (16)	N6—Cr1—N3	88.04 (17)
N4—Cr1—N6	177.12 (17)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···S3	0.82	2.45	3.234 (5)	161
O2—H2···N7	0.82	2.41	2.840 (9)	114
O2—H2···N8	0.82	1.81	2.536 (8)	147

Data collection: *APEX2* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2044).

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supplementary materials

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2-(2-Hydroxy-1-naphthyldiazenyl)pyridinium [1-(2-pyridyldiazenyl)-2-naphtholato-κ^3N,N',O]tris(thiocyanato-κN)chromium(III) methanol solvate

T.-F. Liu, H.-Y. Lv, B.-T. Zhao and J.-G. Wang

Comment

The color reactions of metal ions and PAN (1-(pyridin-2-yldiazenyl)naphthalen-2-ol) were a useful method in spectrophotometric determination. We report herein the crystal structure of a chromium(III) complex where the Cr^{III} ion is coordinated by a PAN^{-} and three thiocyanate ions.

The crystal structure of (I) consists of a $[Cr(PAN)(NCS)_3]^{-}$ anion, an uncoordinated protonated PAN cation and a methanol molecule (Fig. 1). The chromium ion has a slightly distorted octahedral coordination geometry, its symmetry being close to C_{2V}. The Cr—$N_{(PAN)}$ bond distances are 2.006 (4) and 2.058 (5) Å, the Cr—O bond distance is 1.970 (4) Å. The ligand is planar, the dihedral angle between two aromatic rings is 6.5 (3)°. The Cr—$N_{(NCS)}$ bond distances vary in the range 1.981 (6)–2.001 (6) Å, and the thiocyanate ligands are almost linear with N–C–S angles 178.6 (6)–179.4 (7)°.

The uncoordinated PAN is protonated at (N9), the dihedral angle between two aromatic rings is 15.4 (2)°. Related aromatic rings A and B^{i} [ring A: C5, C6, C7, C8, C9 and C10; ring B: N9, C29, C30, C31, C32 and C33; symmetry code: (i) 1 − *x*, *y*, 1 − *z*) show π-π stacking with a ring-ring separation of 3.594 (4) Å. A C—S···*Cg* (π-ring centre) interaction from the C17—S2 bond to ring R^{ii} [ring *R*: C19, C20, C21, C22, C23 and C28; symmetry code: (ii) −*x*, 1 − *y*, 1 − *z*) is also obsered [C17···*Cg* = 3.684 (7), S···*Cg* = 3.483 (5) Å and C17—S···*Cg* = 84.1 (2)°] (Fig. 2). The crystal packing is characterized by three conventional hydrogen bonds, c.f. Table (Spek, 2003).

Experimental

A methanol solution of PAN (0.3 mmol, 3 ml) was mixed with a methanol solution of chromium(III) nitrate (0.3 mmol, 3 ml). The resulting solution was kept under stirring for about 30 min, and then an aqueous solution of KSCN (0.6 mmol, 6 ml) was added to the mixture. Dark green single crystals were obtained after three weeks (yield 32%).

Refinement

The hydrogen atoms were assigned with isotropic displacement factors and included in the final refinement cycles by use of geometrical restraints (C_{ar}–H = 0.93 Å; C_{Me}–H = 0.96 Å; N–H = 0.86 Å; O–H = 0.82 Å).

Figures

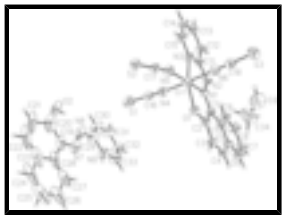

Fig. 1. *ORTEP* plot of $[Cr(C_{10}H_6NO_2)_2(NCS)(C_2H_6O)]$; displacement ellipsoids are drawn at the 30% probability, and hydrogen atoms are shown as spheres of arbitrary radii.

Fig. 2. The π-π stacking interactions between PAN ligands and C—S ring contacts.

2-(2-Hydroxy-1-naphthyldiazenyl)pyridinium [1-(2-pyridyldiazenyl)-2-naphtholato-κ^3N,N',O]tris(thiocyanato-κN)chromium(III) methanol solvate

Crystal data

$(C_{15}H_{12}N_3O)[Cr(C_{15}H_{10}N_3O)(NCS)_3]\cdot CH_4O$	$Z = 2$
$M_r = 756.82$	$F_{000} = 778$
Triclinic, $P\overline{1}$	$D_x = 1.435$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.5045$ (11) Å	Cell parameters from 943 reflections
$b = 12.3189$ (11) Å	$\theta = 2.4$–18.5º
$c = 12.5197$ (12) Å	$\mu = 0.56$ mm^{-1}
$\alpha = 95.568$ (2)º	$T = 273$ (2) K
$\beta = 91.851$ (2)º	Block, dark green
$\gamma = 96.973$ (2)º	0.25 × 0.22 × 0.17 mm
$V = 1751.1$ (3) Å^3	

Data collection

Bruker SMART APEX II CCD diffractometer	7770 independent reflections
Radiation source: fine-focus sealed tube	2997 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.039$
$T = 291$(2) K	$\theta_{max} = 27.5$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -15\rightarrow10$
$T_{min} = 0.874$, $T_{max} = 0.912$	$k = -16\rightarrow16$
11793 measured reflections	$l = -16\rightarrow16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.209$

$S = 0.96$

7770 reflections

454 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.30$ e Å^{-3}

$\Delta\rho_{min} = -0.40$ e Å^{-3}

Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cr1	0.67707 (7)	0.78088 (6)	0.54026 (6)	0.0578 (3)
C1	0.4376 (4)	0.7422 (4)	0.4627 (4)	0.0548 (12)
C2	0.4604 (4)	0.6584 (4)	0.5301 (4)	0.0559 (13)
C3	0.3659 (5)	0.5775 (4)	0.5483 (4)	0.0677 (15)
H3	0.3784	0.5222	0.5913	0.081*
C4	0.2590 (5)	0.5803 (4)	0.5038 (5)	0.0727 (16)
H4	0.1985	0.5276	0.5190	0.087*
C5	0.2331 (4)	0.6610 (4)	0.4336 (5)	0.0665 (15)
C6	0.1223 (5)	0.6589 (5)	0.3868 (6)	0.0886 (19)
H6	0.0619	0.6078	0.4051	0.106*
C7	0.0996 (5)	0.7317 (5)	0.3128 (6)	0.102 (2)
H7	0.0250	0.7287	0.2807	0.122*
C8	0.1902 (5)	0.8092 (5)	0.2875 (6)	0.100 (2)
H8	0.1760	0.8576	0.2374	0.120*
C9	0.2999 (4)	0.8154 (4)	0.3350 (5)	0.0741 (16)
H9A	0.3587	0.8689	0.3180	0.089*
C10	0.3250 (4)	0.7413 (4)	0.4100 (4)	0.0620 (14)
C11	0.6506 (4)	0.9631 (4)	0.4199 (4)	0.0536 (12)
C12	0.6706 (5)	1.0577 (4)	0.3700 (4)	0.0674 (15)
H12	0.6115	1.0803	0.3281	0.081*
C13	0.7799 (5)	1.1187 (4)	0.3829 (5)	0.0785 (17)
H13	0.7958	1.1828	0.3495	0.094*
C14	0.8649 (5)	1.0833 (5)	0.4458 (5)	0.0891 (19)
H14	0.9392	1.1229	0.4549	0.107*
C15	0.8397 (4)	0.9896 (5)	0.4951 (5)	0.0799 (17)
H15	0.8968	0.9684	0.5402	0.096*
C16	0.7459 (4)	0.6619 (4)	0.3243 (5)	0.0631 (14)
C17	0.8993 (4)	0.7003 (4)	0.6412 (4)	0.0530 (12)
C18	0.6145 (4)	0.9187 (5)	0.7523 (5)	0.0639 (14)
C19	0.9169 (7)	0.2356 (8)	1.0362 (5)	0.106 (3)
C20	1.0369 (13)	0.2950 (12)	1.0400 (8)	0.156 (6)
C21	1.1309 (12)	0.2374 (15)	1.0798 (11)	0.221 (10)
H21	1.2080	0.2703	1.0786	0.265*
C22	1.1116 (19)	0.1451 (17)	1.1155 (14)	0.244 (14)

H22	1.1756	0.1118	1.1369	0.293*
C23	0.9927 (12)	0.0864 (11)	1.1256 (7)	0.171 (5)
C24	0.9728 (15)	−0.0160 (12)	1.1704 (12)	0.214 (10)
H24	1.0357	−0.0445	1.2011	0.256*
C25	0.8660 (16)	−0.0715 (12)	1.1694 (12)	0.223 (10)
H25	0.8551	−0.1393	1.1969	0.268*
C26	0.7713 (10)	−0.0280 (7)	1.1273 (6)	0.157 (4)
H26	0.6962	−0.0657	1.1276	0.188*
C27	0.7892 (10)	0.0735 (7)	1.0842 (6)	0.130 (4)
H27	0.7253	0.1026	1.0561	0.156*
C28	0.8979 (9)	0.1299 (9)	1.0829 (6)	0.120 (4)
C29	0.7445 (5)	0.3957 (6)	0.8938 (4)	0.0848 (18)
C30	0.7542 (5)	0.4775 (6)	0.8238 (4)	0.097 (2)
H30	0.8272	0.5149	0.8122	0.116*
C31	0.6560 (7)	0.5021 (6)	0.7725 (5)	0.105 (2)
H31	0.6620	0.5569	0.7261	0.126*
C32	0.5476 (6)	0.4462 (6)	0.7889 (6)	0.102 (2)
H32	0.4804	0.4622	0.7534	0.123*
C33	0.5414 (6)	0.3685 (6)	0.8571 (5)	0.103 (2)
H33	0.4689	0.3305	0.8695	0.124*
C34	0.4037 (9)	0.7067 (6)	0.8293 (7)	0.171 (4)
H34A	0.3448	0.7471	0.8023	0.256*
H34B	0.3691	0.6336	0.8392	0.256*
H34C	0.4647	0.7031	0.7790	0.256*
N1	0.5352 (3)	0.8163 (3)	0.4601 (3)	0.0515 (10)
N2	0.5393 (3)	0.9016 (3)	0.4073 (3)	0.0543 (10)
N3	0.7350 (3)	0.9268 (3)	0.4807 (3)	0.0617 (11)
N4	0.7172 (4)	0.7007 (4)	0.4042 (4)	0.0668 (12)
N5	0.8219 (4)	0.7427 (4)	0.6115 (3)	0.0690 (12)
N6	0.6368 (4)	0.8688 (4)	0.6740 (4)	0.0663 (12)
N7	0.8267 (4)	0.2708 (5)	0.9922 (4)	0.0939 (17)
N8	0.8414 (4)	0.3668 (6)	0.9466 (4)	0.0974 (18)
N9	0.6382 (4)	0.3447 (4)	0.9076 (4)	0.0877 (15)
H9	0.6312	0.2944	0.9509	0.105*
O1	0.5634 (3)	0.6576 (3)	0.5728 (3)	0.0601 (9)
O2	1.0568 (5)	0.3861 (8)	1.0048 (5)	0.158 (4)
H2	0.9979	0.3994	0.9724	0.236*
O3	0.4523 (5)	0.7608 (5)	0.9300 (4)	0.1310 (18)
H3A	0.5003	0.8131	0.9193	0.196*
S1	0.78500 (16)	0.60433 (18)	0.21174 (16)	0.1208 (7)
S2	1.00656 (11)	0.64120 (11)	0.68460 (12)	0.0674 (4)
S3	0.58372 (16)	0.98911 (14)	0.86114 (13)	0.0944 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr1	0.0502 (5)	0.0543 (5)	0.0720 (6)	0.0090 (4)	−0.0011 (4)	0.0206 (4)
C1	0.047 (3)	0.048 (3)	0.071 (3)	0.000 (2)	0.001 (3)	0.021 (3)

C2	0.053 (3)	0.048 (3)	0.068 (3)	0.006 (2)	0.001 (3)	0.013 (3)
C3	0.072 (4)	0.049 (3)	0.085 (4)	0.002 (3)	0.018 (3)	0.025 (3)
C4	0.068 (4)	0.047 (3)	0.102 (4)	−0.008 (3)	0.016 (3)	0.014 (3)
C5	0.049 (3)	0.046 (3)	0.101 (4)	−0.006 (2)	−0.002 (3)	0.007 (3)
C6	0.055 (4)	0.064 (4)	0.141 (6)	−0.010 (3)	−0.013 (4)	0.008 (4)
C7	0.057 (4)	0.084 (5)	0.160 (7)	−0.001 (4)	−0.028 (4)	0.014 (5)
C8	0.059 (4)	0.086 (4)	0.151 (6)	−0.011 (3)	−0.043 (4)	0.032 (4)
C9	0.054 (3)	0.058 (3)	0.109 (4)	−0.005 (3)	−0.022 (3)	0.025 (3)
C10	0.051 (3)	0.044 (3)	0.090 (4)	−0.001 (2)	−0.001 (3)	0.012 (3)
C11	0.044 (3)	0.044 (3)	0.073 (3)	0.000 (2)	−0.002 (3)	0.017 (3)
C12	0.059 (3)	0.054 (3)	0.090 (4)	−0.002 (3)	−0.007 (3)	0.024 (3)
C13	0.065 (4)	0.056 (3)	0.115 (5)	−0.003 (3)	0.008 (4)	0.022 (3)
C14	0.047 (3)	0.070 (4)	0.149 (6)	−0.014 (3)	0.001 (4)	0.034 (4)
C15	0.040 (3)	0.078 (4)	0.124 (5)	0.002 (3)	−0.008 (3)	0.031 (4)
C16	0.049 (3)	0.064 (4)	0.077 (4)	−0.002 (3)	0.001 (3)	0.021 (3)
C17	0.051 (3)	0.052 (3)	0.058 (3)	0.003 (2)	0.005 (3)	0.018 (2)
C18	0.052 (3)	0.064 (4)	0.077 (4)	0.005 (3)	−0.012 (3)	0.021 (3)
C19	0.092 (5)	0.167 (8)	0.062 (4)	0.069 (6)	−0.024 (4)	−0.034 (5)
C20	0.132 (10)	0.257 (16)	0.081 (7)	0.087 (12)	−0.018 (7)	−0.046 (8)
C21	0.125 (9)	0.38 (2)	0.150 (11)	0.169 (15)	−0.083 (8)	−0.158 (14)
C22	0.234 (16)	0.34 (3)	0.167 (13)	0.24 (2)	−0.118 (12)	−0.149 (16)
C23	0.204 (11)	0.199 (12)	0.117 (7)	0.153 (10)	−0.081 (8)	−0.079 (7)
C24	0.34 (2)	0.171 (15)	0.145 (9)	0.201 (17)	−0.130 (14)	−0.068 (10)
C25	0.41 (3)	0.142 (12)	0.126 (8)	0.147 (16)	−0.101 (16)	−0.042 (8)
C26	0.263 (12)	0.109 (7)	0.111 (6)	0.100 (8)	−0.041 (7)	−0.006 (5)
C27	0.209 (10)	0.114 (7)	0.078 (5)	0.100 (7)	−0.050 (6)	−0.018 (5)
C28	0.134 (7)	0.156 (9)	0.079 (5)	0.108 (7)	−0.045 (5)	−0.036 (5)
C29	0.067 (4)	0.127 (5)	0.059 (4)	−0.009 (4)	−0.006 (3)	0.033 (4)
C30	0.071 (4)	0.150 (6)	0.064 (4)	−0.029 (4)	−0.012 (3)	0.042 (4)
C31	0.116 (6)	0.119 (6)	0.077 (4)	−0.017 (5)	−0.032 (4)	0.050 (4)
C32	0.076 (5)	0.121 (6)	0.115 (5)	0.003 (4)	−0.021 (4)	0.058 (5)
C33	0.074 (4)	0.118 (6)	0.121 (5)	0.005 (4)	−0.019 (4)	0.054 (5)
C34	0.278 (13)	0.092 (6)	0.141 (8)	0.003 (7)	0.079 (9)	0.009 (6)
N1	0.048 (2)	0.046 (2)	0.064 (2)	0.0084 (19)	0.002 (2)	0.016 (2)
N2	0.048 (2)	0.043 (2)	0.072 (3)	−0.0033 (18)	−0.009 (2)	0.023 (2)
N3	0.043 (2)	0.059 (3)	0.084 (3)	0.002 (2)	−0.005 (2)	0.021 (2)
N4	0.064 (3)	0.063 (3)	0.076 (3)	0.010 (2)	0.013 (3)	0.018 (3)
N5	0.057 (3)	0.073 (3)	0.082 (3)	0.019 (2)	−0.003 (2)	0.025 (2)
N6	0.062 (3)	0.064 (3)	0.075 (3)	0.013 (2)	−0.004 (3)	0.013 (3)
N7	0.082 (4)	0.149 (5)	0.058 (3)	0.045 (4)	−0.012 (3)	0.011 (3)
N8	0.065 (3)	0.169 (6)	0.062 (3)	0.021 (4)	−0.012 (3)	0.032 (4)
N9	0.072 (3)	0.113 (4)	0.083 (3)	0.001 (3)	−0.006 (3)	0.053 (3)
O1	0.057 (2)	0.052 (2)	0.075 (2)	0.0083 (17)	0.0027 (18)	0.0236 (17)
O2	0.050 (3)	0.331 (12)	0.083 (4)	0.039 (6)	−0.017 (3)	−0.034 (5)
O3	0.162 (5)	0.117 (4)	0.122 (4)	0.006 (4)	0.041 (4)	0.059 (4)
S1	0.0898 (13)	0.1460 (18)	0.1114 (14)	−0.0141 (12)	0.0249 (12)	−0.0339 (13)
S2	0.0527 (8)	0.0620 (9)	0.0906 (10)	0.0118 (7)	−0.0018 (7)	0.0200 (8)
S3	0.1116 (14)	0.0906 (12)	0.0783 (11)	0.0090 (10)	0.0017 (10)	0.0002 (9)

Geometric parameters (Å, °)

Cr1—O1	1.964 (3)	C18—S3	1.616 (6)
Cr1—N4	1.981 (5)	C19—N7	1.299 (7)
Cr1—N5	1.992 (4)	C19—C28	1.473 (12)
Cr1—N1	2.003 (4)	C19—C20	1.479 (15)
Cr1—N6	2.003 (5)	C20—O2	1.244 (13)
Cr1—N3	2.058 (4)	C20—C21	1.463 (15)
C1—N1	1.362 (5)	C21—C22	1.26 (3)
C1—C10	1.433 (6)	C21—H21	0.9300
C1—C2	1.438 (6)	C22—C23	1.48 (2)
C2—O1	1.286 (5)	C22—H22	0.9300
C2—C3	1.422 (6)	C23—C28	1.387 (10)
C3—C4	1.339 (7)	C23—C24	1.43 (2)
C3—H3	0.9300	C24—C25	1.33 (2)
C4—C5	1.440 (7)	C24—H24	0.9300
C4—H4	0.9300	C25—C26	1.382 (14)
C5—C6	1.381 (7)	C25—H25	0.9300
C5—C10	1.416 (6)	C26—C27	1.404 (11)
C6—C7	1.391 (8)	C26—H26	0.9300
C6—H6	0.9300	C27—C28	1.356 (12)
C7—C8	1.392 (8)	C27—H27	0.9300
C7—H7	0.9300	C29—N9	1.330 (7)
C8—C9	1.368 (7)	C29—N8	1.379 (7)
C8—H8	0.9300	C29—C30	1.395 (8)
C9—C10	1.419 (7)	C30—C31	1.362 (8)
C9—H9A	0.9300	C30—H30	0.9300
C11—N3	1.358 (5)	C31—C32	1.381 (8)
C11—C12	1.374 (6)	C31—H31	0.9300
C11—N2	1.403 (5)	C32—C33	1.341 (8)
C12—C13	1.380 (7)	C32—H32	0.9300
C12—H12	0.9300	C33—N9	1.340 (7)
C13—C14	1.374 (7)	C33—H33	0.9300
C13—H13	0.9300	C34—O3	1.430 (9)
C14—C15	1.366 (7)	C34—H34A	0.9600
C14—H14	0.9300	C34—H34B	0.9600
C15—N3	1.346 (6)	C34—H34C	0.9600
C15—H15	0.9300	N1—N2	1.292 (4)
C16—N4	1.143 (6)	N7—N8	1.356 (7)
C16—S1	1.617 (6)	N9—H9	0.8600
C17—N5	1.155 (5)	O2—H2	0.8200
C17—S2	1.612 (5)	O3—H3A	0.8200
C18—N6	1.159 (6)		
O1—Cr1—N4	92.38 (16)	O2—C20—C21	122.2 (16)
O1—Cr1—N5	100.34 (15)	O2—C20—C19	121.2 (12)
N4—Cr1—N5	89.61 (17)	C21—C20—C19	116.5 (13)
O1—Cr1—N1	80.57 (14)	C22—C21—C20	123 (2)
N4—Cr1—N1	87.03 (16)	C22—C21—H21	118.7

N5—Cr1—N1	176.56 (16)	C20—C21—H21	118.7
O1—Cr1—N6	89.83 (16)	C21—C22—C23	123.9 (15)
N4—Cr1—N6	177.12 (17)	C21—C22—H22	118.0
N5—Cr1—N6	91.81 (17)	C23—C22—H22	118.0
N1—Cr1—N6	91.51 (16)	C28—C23—C24	118.9 (15)
O1—Cr1—N3	157.01 (15)	C28—C23—C22	118.1 (14)
N4—Cr1—N3	89.21 (17)	C24—C23—C22	122.9 (12)
N5—Cr1—N3	102.61 (16)	C25—C24—C23	121.4 (15)
N1—Cr1—N3	76.61 (15)	C25—C24—H24	119.3
N6—Cr1—N3	88.04 (17)	C23—C24—H24	119.3
N1—C1—C10	128.6 (4)	C24—C25—C26	119.7 (17)
N1—C1—C2	110.1 (4)	C24—C25—H25	120.1
C10—C1—C2	121.3 (4)	C26—C25—H25	120.1
O1—C2—C3	121.4 (4)	C25—C26—C27	119.6 (13)
O1—C2—C1	120.5 (4)	C25—C26—H26	120.2
C3—C2—C1	118.1 (5)	C27—C26—H26	120.2
C4—C3—C2	120.5 (5)	C28—C27—C26	121.3 (10)
C4—C3—H3	119.8	C28—C27—H27	119.4
C2—C3—H3	119.8	C26—C27—H27	119.4
C3—C4—C5	123.2 (5)	C27—C28—C23	119.1 (12)
C3—C4—H4	118.4	C27—C28—C19	121.3 (7)
C5—C4—H4	118.4	C23—C28—C19	119.6 (11)
C6—C5—C10	120.1 (5)	N9—C29—N8	120.4 (5)
C6—C5—C4	121.3 (5)	N9—C29—C30	117.9 (6)
C10—C5—C4	118.6 (5)	N8—C29—C30	121.7 (6)
C5—C6—C7	121.3 (6)	C31—C30—C29	119.5 (6)
C5—C6—H6	119.4	C31—C30—H30	120.2
C7—C6—H6	119.4	C29—C30—H30	120.2
C6—C7—C8	118.9 (6)	C30—C31—C32	120.4 (6)
C6—C7—H7	120.6	C30—C31—H31	119.8
C8—C7—H7	120.6	C32—C31—H31	119.8
C9—C8—C7	121.1 (6)	C33—C32—C31	118.5 (6)
C9—C8—H8	119.4	C33—C32—H32	120.8
C7—C8—H8	119.4	C31—C32—H32	120.8
C8—C9—C10	120.8 (5)	N9—C33—C32	120.9 (6)
C8—C9—H9A	119.6	N9—C33—H33	119.6
C10—C9—H9A	119.6	C32—C33—H33	119.6
C5—C10—C9	117.8 (5)	O3—C34—H34A	109.5
C5—C10—C1	118.1 (4)	O3—C34—H34B	109.5
C9—C10—C1	124.0 (4)	H34A—C34—H34B	109.5
N3—C11—C12	122.3 (4)	O3—C34—H34C	109.5
N3—C11—N2	118.9 (4)	H34A—C34—H34C	109.5
C12—C11—N2	118.8 (4)	H34B—C34—H34C	109.5
C11—C12—C13	118.8 (5)	N2—N1—C1	123.5 (4)
C11—C12—H12	120.6	N2—N1—Cr1	121.4 (3)
C13—C12—H12	120.6	C1—N1—Cr1	115.1 (3)
C14—C13—C12	119.0 (5)	N1—N2—C11	110.8 (4)
C14—C13—H13	120.5	C15—N3—C11	117.7 (4)
C12—C13—H13	120.5	C15—N3—Cr1	130.0 (4)

C15—C14—C13	119.7 (5)	C11—N3—Cr1	112.2 (3)
C15—C14—H14	120.1	C16—N4—Cr1	174.6 (5)
C13—C14—H14	120.1	C17—N5—Cr1	166.2 (4)
N3—C15—C14	122.3 (5)	C18—N6—Cr1	179.0 (4)
N3—C15—H15	118.9	C19—N7—N8	119.3 (7)
C14—C15—H15	118.9	N7—N8—C29	116.7 (5)
N4—C16—S1	178.7 (5)	C29—N9—C33	122.8 (5)
N5—C17—S2	179.1 (5)	C29—N9—H9	118.6
N6—C18—S3	179.6 (6)	C33—N9—H9	118.6
N7—C19—C28	117.8 (8)	C2—O1—Cr1	113.7 (3)
N7—C19—C20	123.4 (10)	C20—O2—H2	109.5
C28—C19—C20	118.8 (8)	C34—O3—H3A	109.5
N1—C1—C2—O1	−3.8 (6)	C29—C30—C31—C32	−0.4 (11)
C10—C1—C2—O1	176.8 (4)	C30—C31—C32—C33	0.7 (11)
N1—C1—C2—C3	175.9 (4)	C31—C32—C33—N9	−0.5 (11)
C10—C1—C2—C3	−3.4 (7)	C10—C1—N1—N2	0.6 (8)
O1—C2—C3—C4	179.4 (5)	C2—C1—N1—N2	−178.7 (4)
C1—C2—C3—C4	−0.3 (8)	C10—C1—N1—Cr1	−177.0 (4)
C2—C3—C4—C5	2.0 (8)	C2—C1—N1—Cr1	3.7 (5)
C3—C4—C5—C6	178.0 (5)	O1—Cr1—N1—N2	−180.0 (4)
C3—C4—C5—C10	0.1 (8)	N4—Cr1—N1—N2	−87.1 (3)
C10—C5—C6—C7	2.7 (9)	N6—Cr1—N1—N2	90.4 (4)
C4—C5—C6—C7	−175.1 (6)	N3—Cr1—N1—N2	2.8 (3)
C5—C6—C7—C8	−1.2 (10)	O1—Cr1—N1—C1	−2.3 (3)
C6—C7—C8—C9	−0.8 (10)	N4—Cr1—N1—C1	90.6 (3)
C7—C8—C9—C10	1.3 (10)	N6—Cr1—N1—C1	−91.9 (3)
C6—C5—C10—C9	−2.1 (8)	N3—Cr1—N1—C1	−179.5 (4)
C4—C5—C10—C9	175.8 (5)	C1—N1—N2—C11	179.7 (4)
C6—C5—C10—C1	178.4 (5)	Cr1—N1—N2—C11	−2.9 (5)
C4—C5—C10—C1	−3.8 (7)	N3—C11—N2—N1	0.9 (6)
C8—C9—C10—C5	0.1 (8)	C12—C11—N2—N1	−179.4 (4)
C8—C9—C10—C1	179.6 (5)	C14—C15—N3—C11	−4.1 (8)
N1—C1—C10—C5	−173.8 (5)	C14—C15—N3—Cr1	177.6 (4)
C2—C1—C10—C5	5.4 (7)	C12—C11—N3—C15	2.9 (7)
N1—C1—C10—C9	6.7 (9)	N2—C11—N3—C15	−177.4 (5)
C2—C1—C10—C9	−174.1 (5)	C12—C11—N3—Cr1	−178.5 (4)
N3—C11—C12—C13	−0.7 (8)	N2—C11—N3—Cr1	1.2 (5)
N2—C11—C12—C13	179.6 (5)	O1—Cr1—N3—C15	169.4 (4)
C11—C12—C13—C14	−0.4 (9)	N4—Cr1—N3—C15	−96.4 (5)
C12—C13—C14—C15	−0.6 (9)	N5—Cr1—N3—C15	−7.0 (5)
C13—C14—C15—N3	3.0 (10)	N1—Cr1—N3—C15	176.4 (5)
N7—C19—C20—O2	3.4 (16)	N6—Cr1—N3—C15	84.4 (5)
C28—C19—C20—O2	−177.7 (10)	O1—Cr1—N3—C11	−9.0 (6)
N7—C19—C20—C21	−172.2 (8)	N4—Cr1—N3—C11	85.2 (3)
C28—C19—C20—C21	6.6 (12)	N5—Cr1—N3—C11	174.6 (3)
O2—C20—C21—C22	−180.0 (17)	N1—Cr1—N3—C11	−1.9 (3)
C19—C20—C21—C22	−4(2)	N6—Cr1—N3—C11	−94.0 (3)
C20—C21—C22—C23	−2(3)	O1—Cr1—N5—C17	50.8 (17)
C21—C22—C23—C28	7(2)	N4—Cr1—N5—C17	−41.6 (16)

C21—C22—C23—C24	−177.3 (15)	N6—Cr1—N5—C17	140.9 (16)
C28—C23—C24—C25	2(2)	N3—Cr1—N5—C17	−130.7 (16)
C22—C23—C24—C25	−173.8 (16)	C28—C19—N7—N8	−178.7 (5)
C23—C24—C25—C26	−2(3)	C20—C19—N7—N8	0.2 (10)
C24—C25—C26—C27	1(2)	C19—N7—N8—C29	175.0 (5)
C25—C26—C27—C28	0.1 (13)	N9—C29—N8—N7	14.2 (9)
C26—C27—C28—C23	−0.4 (12)	C30—C29—N8—N7	−165.0 (6)
C26—C27—C28—C19	179.1 (6)	N8—C29—N9—C33	−179.0 (6)
C24—C23—C28—C27	−0.5 (13)	C30—C29—N9—C33	0.3 (10)
C22—C23—C28—C27	175.3 (10)	C32—C33—N9—C29	0.0 (11)
C24—C23—C28—C19	180.0 (9)	C3—C2—O1—Cr1	−177.8 (4)
C22—C23—C28—C19	−4.2 (13)	C1—C2—O1—Cr1	2.0 (6)
N7—C19—C28—C27	−2.9 (11)	N4—Cr1—O1—C2	−86.4 (3)
C20—C19—C28—C27	178.1 (8)	N5—Cr1—O1—C2	−176.4 (3)
N7—C19—C28—C23	176.6 (6)	N1—Cr1—O1—C2	0.2 (3)
C20—C19—C28—C23	−2.3 (11)	N6—Cr1—O1—C2	91.7 (3)
N9—C29—C30—C31	−0.1 (10)	N3—Cr1—O1—C2	7.2 (6)
N8—C29—C30—C31	179.2 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···S3	0.82	2.45	3.234 (5)	161
O2—H2···N7	0.82	2.41	2.840 (9)	114
O2—H2···N8	0.82	1.81	2.536 (8)	147

Fig. 1

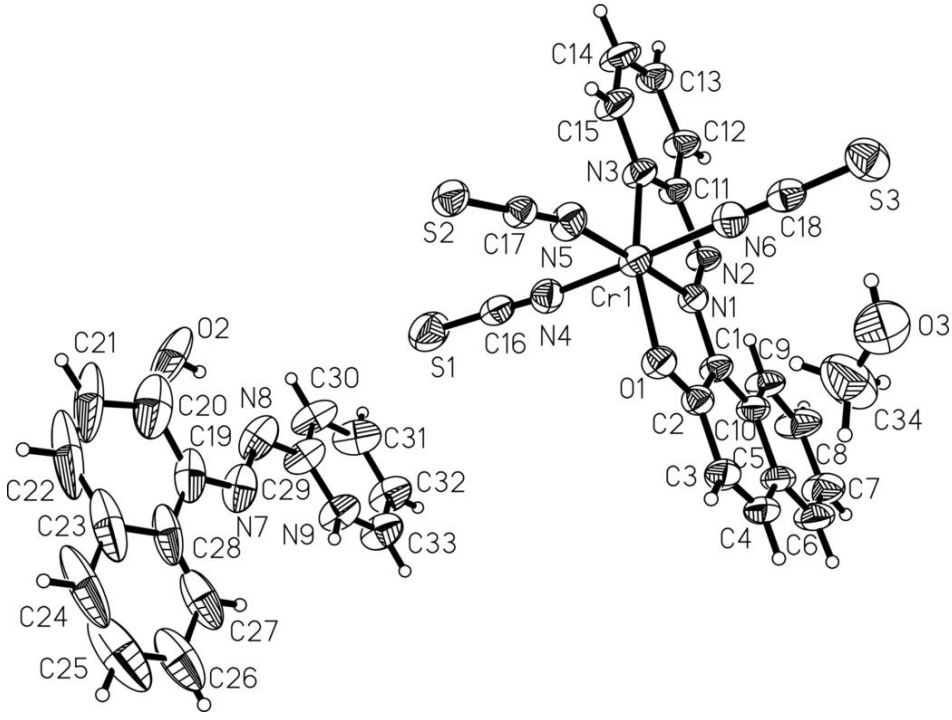
C14
C13
C15
C12
N3
C11
S3
S2
C17
N5
N6
C18
Cr1
N2
N1
C16
N4
S1
C1
C9
O3
O1
C2
C10
C34
C5
C8
C3
C7
C4
C6
C21
O2
C30
N8
C20
C19
C31
C22
C29
C32
C23
N7
N9
C28
C33
C24
C27
C25
C26

Fig. 2