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Chromium(III) Complexes of Macrocyclic Tetraamine Ligands. Crystal Structures of *cis*-Dihydroxo-(*rac*-5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane)chromium(III) Perchlorate Dihydrate and Di-*cis*-carbonato-(*rac*-5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane)chromium(III) Dithionate Tetrahydrate

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cis-$[Cr(cyc\textit{b})(OH)_2]ClO_4 \cdot 2H_2O$, where cyc*b* is the title amine, crystallizes in space group *C*2/*c* with $a=17.260(4)$ Å, $b=10.573(2)$ Å, $c=13.346(3)$ Å, $\beta=96.75(2)°$ and $Z=4$. *cis*-$[Cr(cyc\textit{b})O_2CO]_2S_2O_6 \cdot 4H_2O$ crystallizes in space group $P\bar{1}$ with $a=8.788(1)$ Å, $b=10.748(1)$ Å, $c=14.240(1)$ Å, $\alpha=74.63(1)°$, $\beta=83.70(1)°$, $\gamma=68.06(1)°$ and $Z=1$. Data for 1996 and 3338 observed reflections, $F^2>2\sigma(F^2)$, were refined to give *R* factors of 0.039 and 0.033, respectively, for the two structures. Both chromium(III) cations have a distorted octahedral geometry with the macrocyclic ligand in the same so-called "folded" configuration. The configurations of the 5- and 6-membered rings are *gauche* and *chair*, respectively. The N4 and N11 nitrogen atoms are coordinated in the *trans* position to each other, and the angle of folding is the same in the two cations. The central metal atom is, however, placed differently relative to the amine ligand, and the chromium-nitrogen bond lengths are significantly longer in the dihydroxo than in the carbonato complex ion.

Chromium(III) reacts with *rac*-5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane, cyc*b* *cf.* Fig. 1, to give *cis*-tetraamine complexes which exhibit unusual spectral and kinetic properties compared to other chromium(III) complexes.[1] The *cis*-dihydroxy complex shows a first spin allowed absorption band, which is clearly split into two components, and the unusual robustness of the *cis*-Cr(cyc*b*) moiety has allowed the preparation of a tetraaminecarbonato species under conditions where amine aquation normally takes place. The present investigation was initiated to provide structural data in an attempt to rationalize these observations.

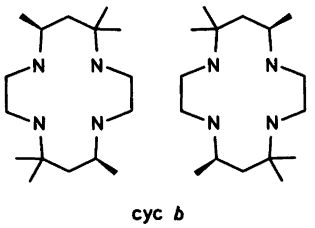

Fig. 1. *rac*-5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane.

RESULTS and DISCUSSION

cis-$[Cr(cyc\textit{b})(OH)_2]ClO_4 \cdot 2H_2O$ and *cis*-$[Cr(cyc\textit{b})O_2CO]_2S_2O_6 \cdot 4H_2O$ both crystallize in centrosymmetric space groups which contain an

0302-4377/84 $2.50

Table 1. Crystal data and data collection and reduction characteristics.

	cis-[Cr(cyc*b*)(OH)$_2$]ClO$_4$·2H$_2$O[a]	*cis*-[Cr(cyc*b*)O$_2$CO]$_2$S$_2$O$_6$·4H$_2$O[a]
Space group	Monoclinic *C2/c*	Triclinic $P\bar{1}$
Cell parameters		
a (Å)	17.260(4)	8.788(1)
b (Å)	10.573(2)	10.748(1)
c (Å)	13.346(3)	14.240(1)
α (°)		74.63(1)
β (°)	96.75(2)	83.70(1)
γ (°)		68.06(1)
M (g mol^{-1})	506.0	1025.2
V (Å^3)	2416.5	1202.8
D_m (g cm^{-3})	1.41	1.41
D_x (g cm^{-3})	1.391	1.415
Z	4	1
F(000)	1084	548
μ(Mo*K*α) (cm^{-1})	6.2	5.9
crystal size (mm^3)	0.13×0.15×0.30	0.09×0.16×0.30
No. of reflections:		
Total, measured	3726	5115
Independent, observed	1996	3338
LT[$F^2<2\sigma(F^2)$]	779	890
No. of parameters	240	441
R	0.039	0.033
1/weight	$\sigma^2(F)-0.025\|F\|+0.0013F^2$	$\sigma^2(F)+0.008\|F\|+0.00006F^2$
R_w	0.051	0.040

[a] cyc*b*≡$C_{16}H_{36}N_4$.

equal number of the two optical isomers, *cf.* Table 1. The dihydroxo compound has the chromium atom on a crystallographic twofold axis, and the carbonato compound is close to having an analogous two-fold symmetry of the cation, and consequently the same configuration of the Cr(III)-cyc*b* moiety, *cf.* Fig. 2. The interatomic bond lengths and angles are very similar in the three independent cyc*b*-ligand halves of the two complex cations, and average

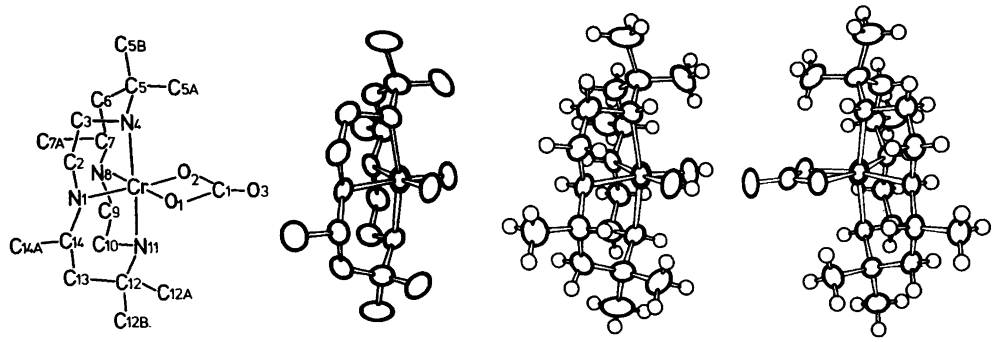

Fig. 2. Numbering scheme, *cis*-[Cr(cyc*b*)(OH)$_2$]$^+$ without and with hydrogen atoms and *cis*-[Cr(cyc*b*)O$_2$CO]$^+$. Hydrogen atoms are drawn as spheres with a radius equivalent to 0.23 Å and the other atoms as 50 % probability ellipsoids.

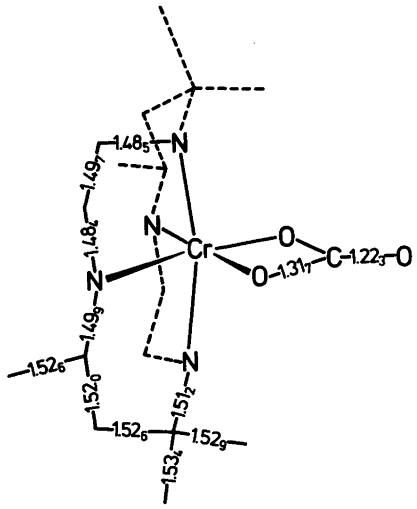

Fig. 3. Average values for bond lengths in Cr(III)-coordinated cyc*b* derived from the 3 independent ligand halves of the dihydroxo- and carbonato-derivatives. Standard deviations in the individual bond lengths are between 0.002 and 0.004 Å, and the maximum deviation of individual bond lengths from the reported mean values are less than 2σ.

values for some selected bond lengths are given in Fig. 3. The chromium-nitrogen bond lengths are more different, as demonstrated in Table 2, and are longer in the dihydroxo- than in the carbonato-cation. This increase in bond length is seen both in the *cis* and *trans* positions relative to the hydroxide ligands.

Both cations show a pronounced tetrahedral distortion of the (CrN_2O_2)-plane. This may be seen in Fig. 2 and is also demonstrated by the data of Table 2, particularly the significant difference between the two types of (N4,N11)-Cr-O angles. This distortion is most likely caused by interactions between this plane and the C5A and C12A methyl groups of the amine ligand, *cf.* Fig. 2, since it is not present to any comparable degree in complexes without these methyl groups as, *e.g.* in *cis*-$[Co(cyclam)(en)]^{3+}$.[2] (cyclam≡1,4,8,11-tetraazacyclotetradecane; en≡1,2-ethanediamine.) This type of distortion rationalizes the red-shift and the increased intensity of the low energy component of the first spin allowed absorption band of *cis*-Cr(cyc*b*)-cations[1] in terms of the generally observed spectroscopic difference between ligand field bands of tetrahedral and octahedral transition metal complexes.

The macrocyclic ligand is folded to make the N4- and N11-nitrogen atoms *trans* to each other, which gives the configuration shown in Fig. 1. This configuration has been predicted to be the most stable,[3] and is also found in a number of other hexacoordinated metal complexes of this ligand. The ligand "folding" is surprisingly constant for the complexes of the different metal ions, and the range of α-values in Table 3, *cf.* Fig. 4, 106.6 to 109.3°, should be compared to the much larger spread of ∠(N1-M-N8)-values, 89.4 to 104.4°. The most significant difference between these structures therefore is the distance δ.

Bond lengths in the coordinated amine can be compared with literature data for the free

Table 2. Comparison of selected metal to ligand bond lengths [Å] and angles [°] in the two Cr(cyc*b*)-cations, *cf.* Fig. 2.[a]

	cis-$[Cr(cyc b)(OH)_2]^+$	*cis*-$[Cr(cyc b)O_2CO]^+$	
Cr–N1,Cr–N8	2.142	2.091	2.106
Cr–N4,Cr–N11	2.140	2.108	2.111
Cr–O1,Cr–O2	1.918	1.945	1.959
N1–Cr–N8	94.85	99.31	
N4–Cr–N11	165.30	169.70	
N1–Cr–N4,N8–Cr–N11	83.32	85.30	84.41
N4–Cr–N8,N1–Cr–N11	86.74	88.45	88.54
O1–Cr–O2	92.15	67.56	
N4–Cr–O1,N11–Cr–O2	88.08	88.94	89.12
N4–Cr–O2,N11–Cr–O1.	102.18	99.35	99.78

[a]Standard deviations in these distances and angles are almost constant, ≈0.002 Å and ≈0.05–0.10°.

Table 3. Folding of cyc*b* ligand, α[°], and central atom position, δ[Å], in some hexacoordinated cyc*b* complexes, *cf.* Fig. 4.

Complex	δ	α	Ref.
$[(cyc\mathit{b})Cr(OH)_2]^+$	0.274(2)	106.6(2)	This work
$[(cyc\mathit{b})Cr(O_2CO)]^{+\,a}$	0.189(2)	107.7(2)	This work
$[(cyc\mathit{b})Co(O_2CR)]^{a,b}$	0.156(6)	109.3(4)	4
$[(cyc\mathit{b})Ni(O_2CCH_3)]^{+\,a}$	0.090(10)	107.3(6)	3
$[(cyc\mathit{b})Ni(O_2CCHOH-$	0.101(5)	108.7(4)	5
$-CHOHCOO)Ni(OH_2)(cyc\mathit{b})]^{a}$	0.203(5)	108.4(4)	
$[(cyc\mathit{b})HgCl_2HgCl_2]$	0.463(7)	106.9(6)	6

[a] α and δ for these complexes, which are without a crystallographic twofold axis, have been calculated by averaging values for the two ligand halves. [b] $-R\equiv-o\text{-}C_6H_4SS\text{-}o\text{-}C_6H_4COO^-$.

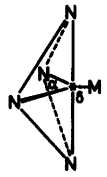

Fig. 4. Ligand folding $\angle$(N-*-N)$\equiv\alpha$, and metal ion position, d(M-*)$\equiv\delta$, of the cyc*b* complexes of Table 4. * is the point of intersection between the *trans*-(N–N) line and the two fold axis.

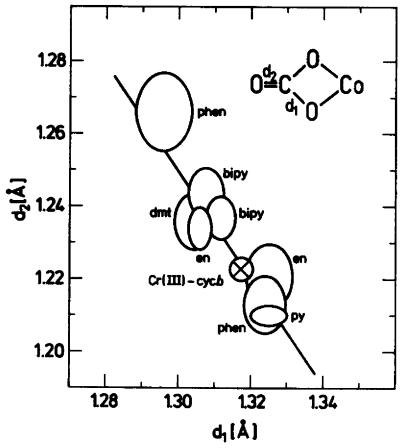

Fig. 5. Comparison of carbon-oxygen bond lengths in *cis*-$[Cr(cyc\mathit{b})O_2CO]^+$ with values for tetraaminecarbonatocobalt(III) complexes. Data and amine ligand name abbreviations are from Ref. 8. Experimental points are indicated by contour ellipses of the probability density functions drawn at the 68 % probability level.

amine,[7] and particularly the carbon-nitrogen bond lengths are found to vary systematically. They increase almost linearly with the number of methyl groups on the carbon atom and increase also when the nitrogen atom is coordinated to Cr(III) or Ni(II).[3,5] The simple empirical expression:

$$d(C-N)\approx 1.46_2+0.01_2 n_{me} + \begin{cases} 0 & [cyc\mathit{b}] \\ 0.02_5 & [Cr(III)\text{-}cyc\mathit{b}] \\ 0.02_1 & [Ni(II)\text{-}cyc\mathit{b}] \end{cases} \text{ Å}$$

where n_{me} is the number of methyl groups on the carbon atom, is thus found to describe accurately the bond length variations of the 44 different carbon-nitrogen bonds of the cyc*b*-amine in Refs. 3, 5, 7 and the present work, *cf.* Fig. 3.

Table 4. Summary of C–H, N–H, and anion bond lengths, [Å], and angles, [°], in *cis*-$[Cr(cyc\mathit{b})(OH)_2](ClO_4)\cdot 2H_2O$ and *cis*-$[Cr(cyc\mathit{b})(O_2CO)]_2(S_2O_6)\cdot 4H_2O$.

	No. of determinations	Range (Std.dev.) [a]
C–H	48	0.83–1.05(3)
N–H	6	0.72–0.77(2)
Cl–O	4	1.325–1.444(8)
O–Cl–O	8	94.8–121.3(6)
S–O	3	1.429–1.435(3)
S–S	1	2.137(1)
O–S–O	3	112.3–114.0(2)
S–S–O	3	105.0–105.4(1)

[a] Standard deviations are almost constant for each bond and angle type.

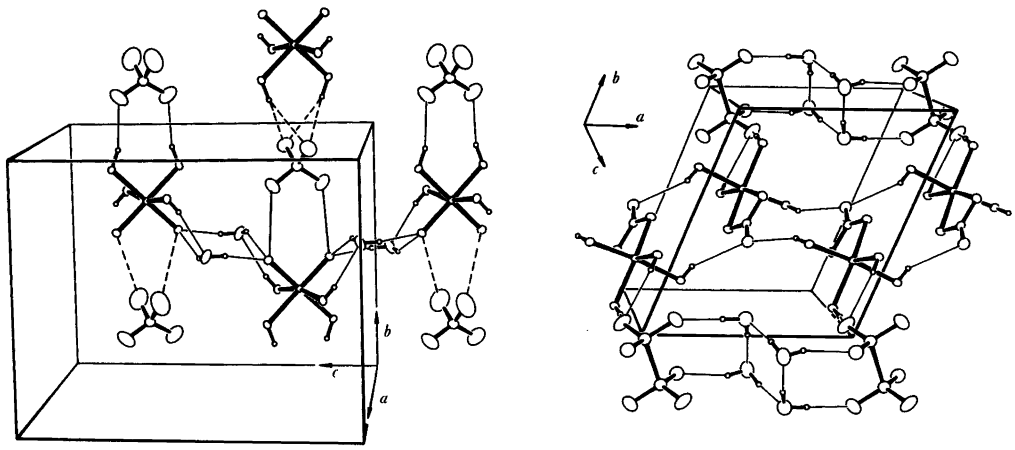

Fig. 6. Two-dimensional net of hydrogen bonds between complex ions, anions, and water molecules in *cis*-[Cr(cyc*b*)(OH)$_2$]ClO$_4$ · 2H$_2$O (left) and in *cis*-[Cr(cyc*b*)O$_2$CO]$_2$S$_2$O$_6$ · 4H$_2$O (right) and orientation relative to the chosen unit cells, *cf.* Table 5 and the text. All carbon atoms and carbon bound hydrogen atoms are omitted. Hydrogen atoms are drawn as spheres with a radius equivalent to 0.10 Å and the other atoms as 35 % probability ellipsoids.

Table 5. Selected intermolecular hydrogen bonds with *d*(A···B) less than 3.3 Å.

AH···B	*d*(H···B) [Å]	*d*(A···B) [Å]
cis-[Cr(cyc*b*)(OH)$_2$]ClO$_4$ · 2H$_2$O:		
OW–H1W···O1	1.91(3)	2.748(3)
OW–H2W···O2	2.50(5)	3.078(4)
O1–(HO1)[a]···O2A		3.239(8)
O1–(HO1)[a]···O3B		>3.3[b]
N1–H1N···O3A	2.68(5)[b]	>3.3[b]
N1–H1N···O3A	2.75(5)[b]	>3.3[b]
N1–H1N···O2B	2.47(3)	3.151(9)
N4–H4N···OW	2.25(3)	3.005(3)
cis-[Cr(cyc*b*)O$_2$CO]$_2$S$_2$O$_6$ · 4H$_2$O:		
O1W–H1WS···O1S	2.02(4)	2.820(4)
O1W–H1WW···O2W	2.07(5)	2.808(4)
O2W–H2WS···O3S	1.89(5)	2.773(5)
O2W–H2WW···O1W	1.98(5)	2.907(5)
N1–H1N···O2S	2.21(2)	2.916(3)
N8–H8N···O2S	2.48(2)	3.203(3)
N4–H4N···O3C	2.35(3)	3.009(3)
N11–H11N···O3C	2.55(3)	3.152(4)

[a] The HO1-atom is not particularly well defined. It is therefore not shown on Fig. 6, and distances involving this atom are not given. [b] These bonds are shown as broken lines on Fig. 6.

Aminecarbonato complexes are easily prepared for a number of transition metal ions and with a variety of amines, and much speculation has been devoted to explain the apparent failure to obtain analogous chromium(III) compounds. The present structure of the complex containing bidentate carbonate shows no anomalies in the chromium-oxygen bond lengths, *cf.* Table 2, and Fig. 5 shows that the bond lengths in the carbonate ligand are similarly influenced by a cobalt(III) center as by a chromium(III) center. The reason why tetraaminecarbonatochromium(III) species have not been characterized previously is most likely kinetically determined as some oxy-anions, including carbonate, for less robust chromium(III) amines are known to catalyze strongly the amine aquation reactions.

Anion bond lengths and angles are within the ranges normally found for perchlorate and dithionate as demonstrated in Table 4. The chlorine atom of the perchlorate ion is placed on a two-fold axis. It has reasonable temperature factors but is surrounded by fractional oxygen atoms to give two equally populated perchlorate ion-positions in the crystal. The dithionate ion is placed to have a centre of symmetry.

Fig. 6 shows the net of intermolecular hydrogen bonds, *cf.* Table 5, in the two crystals. Both compounds are held together by this bond type in

two directions only. These planar sheets are then stacked and held together by interactions between the organic parts of the amine ligand of the cations. The left part of Fig. 6 also shows the two different perchlorate ion orientations in the crystal of the dihydroxy compound.

EXPERIMENTAL

cis-[Cr(cyc*b*)$(OH)_2$]$ClO_4 \cdot 2H_2O$ and *cis*-[Cr(cyc*b*)O_2CO]$Cl \cdot H_2O$ were prepared according to Ref. 1, and the chloride salt of the carbonate complex was converted into the dithionate by precipitation with $Na_2S_2O_6 \cdot 2H_2O$ in aqueous solution to give *cis*-[Cr(cyc*b*)$O_2CO]_2S_2O_6 \cdot 4H_2O$. Analyses: Cr, S, N, C, H. Crystals suitable for the crystallographic work were made by slowly cooling a saturated aqueous solution from 80 °C to room temperature.

The dihydroxy complex forms dark blue monoclinic crystals with tabular habit, showing pleochroism going from yellow to blue in polarized light and with diagonal extinction. The crystals could be cleaved parallel to (001) and (100).

The carbonato complex forms flat, red, triclinic needles which show strong pleochroism from

Table 6. Fractional coordinates for *cis*-[Cr(cyc*b*)$(OH)_2$]$ClO_4 \cdot 2H_2O$.

Atom	X	Y	Z
Cr	.00000	.31750(04)	.25000
O1	.03703(12)	.44333(19)	.34754(14)
N1	.05263(10)	.18043(19)	.35496(12)
N4	.09863(10)	.29160(17)	.16880(14)
C2	.00560(13)	.18601(24)	.44120(16)
C3	.07951(14)	.18486(26)	.09788(18)
C5	.18092(13)	.28435(23)	.22127(17)
C5A	.20139(17)	.41472(27)	.26664(24)
C5B	.23836(20)	.25281(47)	.14567(27)
C6	.18624(14)	.18260(26)	.30276(19)
C7	.13817(13)	.19556(22)	.39006(16)
C7A	.16653(20)	.09868(39)	.47114(27)
Cl	.50000	.32297(09)	.25000
O2A	.4622 (5)	.2487 (7)	.1717 (5)
O3A	.4442 (4)	.4034 (6)	.2915 (5)
O2B	.4579 (6)	.3851 (8)	.1748 (6)
O3B	.4579 (11)	.2382 (14)	.2927 (8)
OW	.39785(18)	.02786(28)	.45579(21)
HO1	.4574 (17)	-.0073 (32)	.1681 (24)
H1N	.0476 (12)	.1182 (24)	.3335 (16)
H2CI	.0173 (12)	.1171 (23)	.4839 (17)
H2CJ	.0230 (14)	.2637 (24)	.4803 (17)
H3CI	.0918 (14)	.0963 (27)	.1302 (19)
H3CJ	.1088 (17)	.1860 (23)	.0497 (22)
H4N	.0969 (14)	.3462 (25)	.1306 (18)
H5AI	.1688 (14)	.4423 (22)	.3110 (20)
H5AJ	.1962 (16)	.4686 (30)	.2123 (22)
H5AK	.2510 (16)	.4137 (26)	.2921 (23)
H5BI	.2339 (19)	.3178 (29)	.0979 (27)
H5BJ	.2313 (23)	.1772 (33)	.1323 (29)
H5BK	.2873 (21)	.2622 (29)	.1759 (23)
H6CI	.2409 (16)	.1747 (19)	.3368 (18)
H6CJ	.1796 (13)	.1055 (24)	.2821 (17)
H7C	.1431 (13)	.2813 (23)	.4141 (17)
H7AI	.1560 (17)	.0096 (35)	.4474 (23)
H7AJ	.2183 (19)	.1033 (26)	.4848 (23)
H7AK	.3514 (19)	.3825 (33)	.4755 (27)
H1W	.4173 (17)	.0290 (29)	.5170 (26)
H2W	.4281 (26)	.0406 (50)	.4292 (39)

Table 7. Fractional coordinates for *cis*-[Cr(cyc*b*)$O_2CO]_2S_2O_6 \cdot 4H_2O$.

Atom	X	Y	Z
Cr	.78757(05)	.37149(04)	.16010(03)
N1	.77500(25)	.17647(19)	.22389(13)
N4	.54153(25)	.44908(20)	.20680(14)
N8	.85784(25)	.41148(20)	.28246(14)
N11	.04216(24)	.27397(19)	.13970(14)
O1C	.70983(21)	.38370(17)	.03403(12)
O2C	.79521(22)	.53568(17)	.06180(12)
O3C	.71506(25)	.56288(21)	-.08935(13)
C1C	.73739(30)	.49919(27)	-.00400(18)
C2	.59618(33)	.20337(26)	.23259(19)
C3	.51122(31)	.32762(26)	.27239(19)
C5	.47526(33)	.57733(26)	.24584(21)
C5A	.46918(39)	.70040(29)	.16086(25)
C5B	.30019(38)	.60005(34)	.28709(27)
C6	.58434(36)	.56311(29)	.32741(22)
C7	.76245(34)	.54801(27)	.30492(20)
C7A	.83515(42)	.56931(36)	.38875(26)
C9	.03529(32)	.38709(26)	.26743(18)
C10	.11757(30)	.25730(26)	.23277(18)
C12	.11000(31)	.14489(24)	.10103(17)
C12A	.05609(37)	.18496(28)	-.00482(18)
C12B	.29767(36)	.09010(29)	.10140(22)
C13	.04847(33)	.03425(24)	.16454(18)
C14	.86461(33)	.06151(24)	.17451(17)
C14A	.83890(39)	-.07196(27)	.23103(20)
S	.08113(10)	.90066(08)	.49174(05)
O1S	.15285(29)	.92553(25)	.39654(14)
O2S	.19600(13)	.85075(29)	.56898(15)
O3S	-.02045(37)	.81961(30)	.50277(25)
O1W	.46852(34)	-.05885(32)	.37318(21)
O2W	.33770(33)	.17794(28)	.45906(20)
H1N	.8043 (28)	.1540 (23)	.2751 (16)
H2CI	.5600 (29)	.2178 (24)	.1722 (17)
H2CJ	.5670 (30)	.1282 (25)	.2667 (18)
H3CI	.5460 (28)	.3119 (23)	.3355 (17)
H3CJ	.4073 (30)	.3385 (25)	.2783 (18)
H4N	.4937 (30)	.4643 (25)	.1625 (18)
H5AI	.5740 (37)	.6959 (31)	.1323 (21)
H5AJ	.3855 (38)	.7120 (32)	.1124 (22)
H5AK	.4280 (43)	.7847 (36)	.1911 (25)
H5BI	.2337 (37)	.5982 (32)	.2443 (22)
H5BJ	.2976 (39)	.5215 (33)	.3491 (23)
H5BK	.2464 (40)	.6879 (34)	.3098 (24)
H6CI	.5350 (35)	.6384 (29)	.3547 (20)
H6CJ	.5794 (32)	.4885 (28)	.3847 (19)
H7C	.7691 (31)	.6112 (26)	.2501 (18)
H7AI	.7614 (43)	.6573 (36)	.4079 (24)
H7AJ	.9416 (37)	.5676 (31)	.3813 (22)
H7AK	.8330 (42)	.5017 (36)	.4412 (24)
H8N	.8510 (27)	.3575 (22)	.3282 (16)
H9CI	.0406 (29)	.4667 (25)	.2209 (18)
H9CJ	.0852 (30)	.3826 (25)	.3236 (18)
H10CI	.2298 (28)	.2420 (23)	.2233 (16)
H10CJ	.1025 (30)	.1836 (25)	.2775 (17)
H11N	.0709 (28)	.3253 (23)	.1023 (16)
H12AI	.1026 (36)	.2492 (30)	-.0428 (21)
H12AJ	.1108 (35)	.1015 (30)	-.0298 (21)
H12AK	.9390 (35)	.2160 (29)	-.0119 (20)
H12BI	.3349 (36)	.0502 (30)	.1677 (22)
H12BJ	.3353 (34)	.0152 (29)	.0718 (21)
H12BK	.3341 (36)	.1598 (31)	.0715 (22)
H13CI	.0939 (30)	.0076 (25)	.2248 (17)
H13CJ	.0957 (29)	-.0386 (24)	.1330 (17)
H14C	.8185 (29)	.0933 (24)	.1119 (17)
H14AI	.7215 (38)	-.0630 (31)	.2300 (22)
H14AJ	.8988 (35)	-.1396 (29)	.1992 (21)
H14AK	.8669 (38)	-.0998 (31)	.3000 (22)
H1WS	.3863 (42)	-.0784 (33)	.3837 (24)
H1WW	.5113 (64)	-.1094 (50)	.4213 (36)
H2WS	.2340 (51)	.1854 (41)	.4873 (30)
H2WW	.3740 (51)	.1062 (42)	.4277 (30)

yellow to red-violet in polarized light and with oblique extinction.

Possible space groups were indicated by Weissenberg and precession photographs. The data were collected at room temperature on an Enraf-Nonius CAD 4 diffractometer with graphite monochromatized Mo$K\alpha$ radiation. Corrections for absorption were not applied. The crystal data are given in Table 1.

Both structures were solved by conventional Patterson and Fourier methods using the XRAY 76 [9] program system. Large temperature factors of the perchlorate oxygen atoms in *cis*-$[Cr(cyc\textit{b})(OH)_2]ClO_4 \cdot 2H_2O$ indicated a possible disorder of this anion, and the final calculations were therefore carried out with four independent oxygen atoms, each given a population of 1/2. Scattering factors for Cr(0) and corrections for anomalous dispersion for Cr, Cl, and S were taken from Ref. 10. Scattering factors for Cl(0), S(0), O(0), N(0), C(0), and H(0) were those of the XRAY 76 system. Weighted least squares refinement with hydrogen atoms having isotropic temperature factors and the other atoms having anisotropic temperature factors gave the agreement factors in Table 1. Positional parameters are given in Tables 6 and 7. Tables of anisotropic temperature factors and F_{obs} are available from the authors upon request.

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