

N4B—H4NF...O8B	0.86	3.11 (2)	145	vii
N5B—H5NB...O9B	0.94	3.09 (2)	168	
N6B—H6NE...O1B	1.02	2.94 (1)	160	vii
N6B—H6NF...O5A	0.63 (1)	3.02 (2)	144	ii

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

Compound (I) was obtained by treating Co(sep)Cl<sub>3</sub> (Bacchi, Ferranti & Pelizzi, 1993) with an equivalent amount of Ag<sub>3</sub>P<sub>3</sub>O<sub>9</sub> in aqueous solution; AgCl was filtered off and after slow evaporation compound (I) precipitated in crystalline form. The structure was solved and refined with standard procedures; all non-H atoms were refined anisotropically. H atoms were localized by difference Fourier maps and were refined isotropically in a full-matrix least-squares cycle except for H1N, H6N and H2O1 which did not show a steady refinement. All H atoms were held fixed in the last least-squares cycles to achieve a better convergence.

Compound (II) was prepared as for (I): a chloride complex obtained by employing a smaller amount of NH<sub>3</sub> and CH<sub>2</sub>O in the step involving the formation of the ligand cage was treated with Ag<sub>3</sub>P<sub>3</sub>O<sub>9</sub>. The observed threshold  $I > 2\sigma(I)$  is used only for calculating  $R_{\text{obs}}$  etc., for comparison with refinements on  $F$ . The refinement was performed with anisotropic thermal parameters for the Co, P, N and O atoms of molecule *A*, for the Co, P2, P3 atoms of molecule *B* and for the O atoms of the water molecules. The remaining atoms were refined isotropically. Methylene hydrogens, H1NC, H1NE, H4NE, H6NE, H101, H142, H151, H152 and H172 were introduced in calculated positions; the remaining amine and water H atoms were located by difference Fourier maps and held fixed in successive least-squares calculations. The  $z$  and  $U_{33}$  parameters are, in general, affected by the largest shift/e.s.d. and, in particular, unstable refinement is observed for the  $z$  coordinate of C3 and C4 of both molecules, probably due to the correlation effects introduced by pseudosymmetry.

Extensive use was made of the Cambridge Structural Database. Calculations were performed on Encore-Gould-Powernode 6040 and Encore 91 computers of the Centro di Studio per la Strutturistica Diffraattometrica del CNR, Parma.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71306 (82 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1051]

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## Structure of Dicarboxyl[(8,9,11,12,13,14- $\eta$ )-12,19-dimethoxy podocarpa-8,11,13-triene]-thiocarbonylchromium(0)

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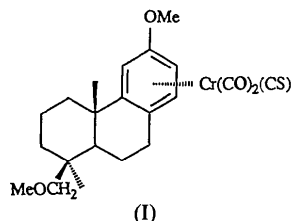
(Received 21 December 1992; accepted 26 April 1993)

### Abstract

The structure determination of the title compound [dicarbonyl( $\eta^6$ -1,2,3,4,4a,9,10,10a-octahydro-6-methoxy-1-methoxymethyl-1,4a-dimethylphenanthrene)-thiocarbonylchromium(0)] establishes that the Cr(CO)<sub>2</sub>(CS) moiety binds to the aromatic ring from the side opposite to that of the methyl group on C12, giving the  $\alpha$  stereoisomer. The carbonyl and thiocarbonyl groups are oriented approximately halfway between the staggered and eclipsed positions.

### Comment

The title compound (I) was prepared during a study of the cyclopentaannulation of diterpenoids mediated by organotransition-metal complexes. In



view of the fact that the preferred conformation of the Cr(CO)<sub>3</sub> moiety in a related diterpenoid tricarbonylchromium complex controlled the regiochemistry of attack of cyano-stabilized carbanions

on the arene ring (Cambie, Erson, Gourdie, Rutledge & Woodgate, 1988), it was of interest to determine the preferred conformation of the  $\text{Cr}(\text{CO})_2\text{CS}$  unit. Although two diastereoisomers are possible, and in fact a mixture of  $\alpha$  and  $\beta$   $\text{Cr}(\text{CO})_3$  complexes was formed from treatment of the enantiopure parent diterpenoid with  $\text{Cr}(\text{CO})_6$ , a single diastereoisomer resulted after conversion of this mixture into the monothiocarbonyl analogue by CO replacement. In the absence of both stereoisomers of the derived thiocarbonyl complex, neither the configuration of the  $\text{Cr}(\text{CO})_2\text{CS}$  moiety relative to the plane of the aromatic ring, nor its preferred orientation, could be deduced unequivocally from NMR data. Therefore, a single-crystal X-ray determination was carried out in order to verify the structure of the complex.

The crystals contain one monomer of complex per asymmetric unit. The molecular geometry and atomic numbering scheme are shown in Fig. 1. The chromium coordinates to the thiocarbonyl and two carbonyl groups and bonds in the  $\eta^6$  mode to the aromatic ring of the podocarpa moiety. The geometry is readily visualized as being a variant of the 'piano stool' arrangement exemplified by benzenetricarbonylchromium (Bailey & Dahl, 1965; Rees & Coppens, 1973). The  $\text{Cr}(\text{CO})_2(\text{CS})$  cluster lies below the aromatic ring (*i.e.* below relative to the methyl group on C10) and the absolute configuration is thus  $\alpha$ . All bonds and angles appear to be within normal ranges. The  $\text{Cr}-\text{C}(\text{aromatic})$  distances range from 2.186 (8) to 2.312 (6) Å. The mean distance of 2.261 Å is slightly longer than the corresponding mean distances of 2.240 and 2.246 Å in the  $\alpha$  and  $\beta$  stereoisomers of the tricarbonyl analogue derived from the related arene methyl 12-methoxypodocarpa-8,11,13-trien-19-oate (Cambie, Clark, Gourdie, Rutledge & Woodgate, 1985), and 2.23 Å in the  $\alpha$  stereoisomer of the corresponding compound lacking the 12-methoxy group, tricarbonyl[(8,9,11,12,13,14- $\eta$ )-methyl podocarpa-8,11,13-trien-19-oate]chromium (Cambie, Clark, Gallagher, Rutledge, Stone & Woodgate, 1988).

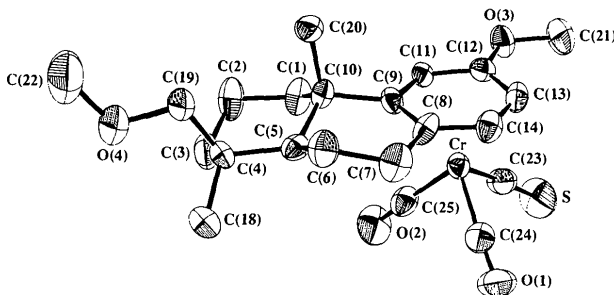


Fig. 1. Molecular geometry and atomic numbering scheme for dicarbonyl[(8,9,11,12,13,14- $\eta$ )-12,19-dimethoxypodocarpa-8,11,13-triene]thiocarbonylchromium(0).

Although the orientation of the  $\pi$ -acceptor ligands relative to the C atoms of the aromatic ring was ambiguous from NMR data, the X-ray structure shows that the carbonyl ligands are oriented approximately halfway between the staggered and eclipsed positions (Fig. 2). This conformation is the same as that found for the  $\alpha$  stereoisomer of tricarbonyl[(8,9,11,12,13,14- $\eta$ )-methyl 12-methoxypodocarpa-8,11,13-trien-19-oate]chromium, but bonds in the  $\beta$  isomer were more nearly eclipsed (Cambie, Clark, Gourdie, Rutledge & Woodgate, 1985). The thiocarbonyl ligand in the present complex is directed towards the C atom of the aromatic ring carrying the methoxy substituent, C12.

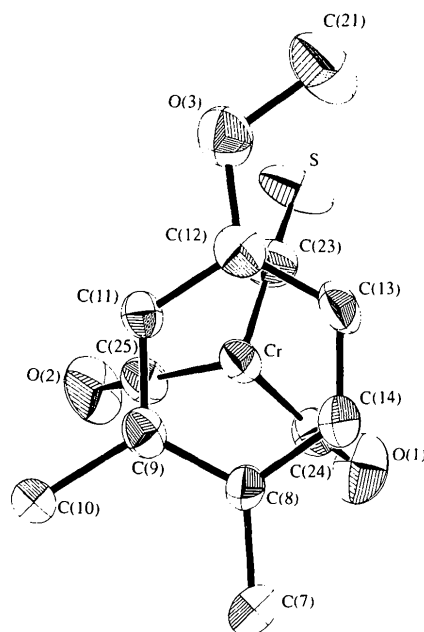


Fig. 2. Relative orientation of the CO and CS groups.

There have been many papers describing the electronic and steric factors responsible for the particular orientation adopted by the  $\text{Cr}(\text{CO})_3$  moiety in  $\text{Cr}(\text{CO})_3(\text{arene})$  complexes. The most recent survey (Hunter, Shilliday, Furey & Zaworotko, 1992) studied the structural manifestations of  $\pi$ -donor and  $\pi$ -acceptor substituents on the arene ring. They deduced that a  $\pi$ -donor substituent on the arene ring would cause the *ipso* C atom and its substituent to bend away from the  $\text{Cr}(\text{CO})_3$  centre with associated loss of arene planarity. The accompanying variations in the  $\text{Cr}-\text{C}(\text{arene})$  distances gave, in their examples, average values of  $\text{Cr}-\text{C}_{ipso}$  2.349,  $-\text{C}_{ortho}$  2.248,  $-\text{C}_{meta}$  2.188 Å. The present complex follows this pattern remarkably closely considering the constraints of the fused ring systems of the podocarpa skeleton. The observed deviation from arene planarity has the *ipso* C12 0.071 Å from the best

plane through the other five arene C atoms in a direction away from the Cr. The observed Cr—C12<sub>ipso</sub>, —C13<sub>ortho</sub> and —C14<sub>meta</sub> distances are 2.302, 2.233 and 2.186 Å, respectively. Similar, though less pronounced effects are present in the  $\alpha$  and  $\beta$  diastereoisomers of tricarbonyl[(8,9,11,12,13,14- $\eta$ )-methyl 12-methoxypodocarpa-8,11,13-trien-19-oate]chromium (Cambie, Clark, Gourdie, Rutledge & Woodgate, 1985).

The Cr—CS, C—S and 2  $\times$  Cr—CO bond lengths of 1.773 (8), 1.573 (8), 1.829 (9) and 1.838 (8) Å, respectively, are normal values. The Cr—CS and C—S values compare well with 1.797 (2) and 1.570 (2) Å in dicarbonyl( $\eta^6$ -methyl benzoate)-thiocarbonylchromium (Saillard, Le Borgne & Grandjean, 1975), and 1.782 (9) and 1.585 (9) Å in dicarbonylthiocarbonyltris(trimethoxyphosphine)-chromium (Bird, Ismail & Butler, 1985).

The shortest intermolecular contact is C12...C18 (3.259 Å), with just two others shorter than 3.5 Å. The molecular packing is shown in Fig. 3.

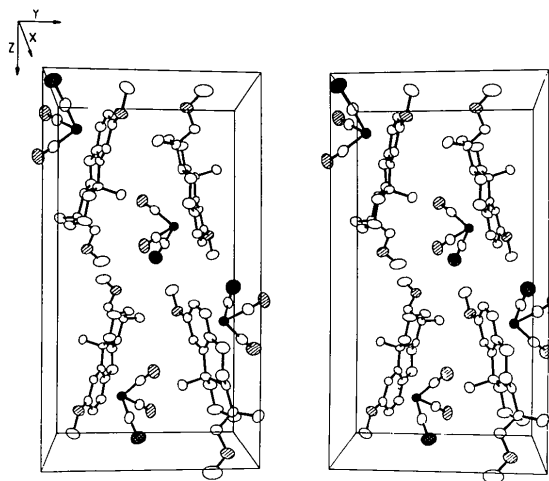


Fig. 3. Stereopair diagrams showing the molecular packing in the unit cell.

## Experimental

### Crystal data

[Cr(C<sub>19</sub>H<sub>28</sub>O<sub>2</sub>)(CO)<sub>2</sub>(CS)]

$M_r = 440.52$

Orthorhombic

$P2_12_12_1$

$a = 7.321$  (6) Å

$b = 12.790$  (1) Å

$c = 22.822$  (3) Å

$V = 2137.0$  (17) Å<sup>3</sup>

$Z = 4$

$D_x = 1.369$  Mg m<sup>-3</sup>

$D_m = 1.33$  (1) Mg m<sup>-3</sup>

Density measured by flotation in aqueous KCl/KI

Mo  $K\alpha$  radiation

$\lambda = 0.71069$  Å

Cell parameters from 25

reflections

$\theta = 9.04$ – $12.67^\circ$

$\mu = 0.658$  mm<sup>-1</sup>

$T = 290.5$  (10) K

Needles

$0.12 \times 0.06 \times 0.04$  mm

Orange

### Data collection

Enraf-Nonius CAD-4 diffractometer

$2\theta/\omega$  scans

Absorption correction:

empirical

$T_{\min} = 0.8985$ ,  $T_{\max} =$

1.0019

2563 measured reflections

2563 independent reflections

1360 observed reflections

$[I > 3\sigma(I)]$

$\theta_{\max} = 27.96^\circ$

$h = 0 \rightarrow 9$

$k = 0 \rightarrow 16$

$l = 0 \rightarrow 30$

3 standard reflections

frequency: 60 min

intensity variation: 4%

### Refinement

Refinement on  $F$

Final  $R = 0.0412$

$wR = 0.0333$

$S = 1.13$

1360 reflections

314 parameters

Only coordinates of H atoms

refined

$w = 1.5735/[\sigma^2(F)$

$+ 0.000127F^2]$

$(\Delta/\sigma)_{\max} = 0.249$  ( $U_{13}$  of O2)

$\Delta\rho_{\max} = 0.32$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.26$  e Å<sup>-3</sup>

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>)

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j B_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	$B_{eq}$
Cr	-0.28311 (14)	-0.63995 (8)	-0.13346 (4)	2.86 (4)
S	-0.6134 (4)	-0.5539 (2)	-0.04445 (13)	7.39 (2)
O1	-0.0649 (9)	-0.4755 (5)	-0.0723 (3)	6.17 (4)
O2	-0.4207 (9)	-0.4721 (5)	-0.2137 (3)	6.64 (4)
O3	-0.5400 (7)	-0.8536 (4)	-0.11338 (18)	3.92 (3)
O4	0.2143 (9)	-0.6752 (4)	-0.4366 (2)	5.95 (4)
C1	-0.3174 (11)	-0.7038 (7)	-0.3062 (3)	4.02 (4)
C2	-0.2816 (13)	-0.6807 (7)	-0.3707 (3)	4.80 (5)
C3	-0.1309 (13)	-0.6020 (7)	-0.3783 (3)	4.49 (5)
C4	0.0515 (10)	-0.6324 (6)	-0.3496 (3)	3.23 (4)
C5	0.0115 (9)	-0.6632 (5)	-0.2847 (3)	2.72 (3)
C6	0.1823 (10)	-0.6973 (7)	-0.2503 (3)	3.79 (4)
C7	0.1467 (10)	-0.6927 (6)	-0.1850 (3)	4.08 (4)
C8	-0.0369 (10)	-0.7312 (6)	-0.1668 (3)	3.06 (4)
C9	-0.1792 (10)	-0.7488 (5)	-0.2071 (3)	2.61 (4)
C10	-0.1460 (9)	-0.7410 (5)	-0.2740 (3)	2.79 (4)
C11	-0.3502 (11)	-0.7842 (5)	-0.1861 (3)	2.45 (3)
C12	-0.3751 (10)	-0.8117 (5)	-0.1267 (3)	3.05 (4)
C13	-0.2347 (12)	-0.7910 (5)	-0.0869 (3)	3.19 (4)
C14	-0.0718 (12)	-0.7515 (7)	-0.1076 (3)	3.29 (4)
C18	0.1733 (11)	-0.5349 (6)	-0.3498 (3)	5.26 (5)
C19	0.1464 (13)	-0.7186 (7)	-0.3844 (3)	4.08 (5)
C20	-0.1056 (12)	-0.8536 (7)	-0.2924 (3)	3.61 (4)
C21	-0.5714 (15)	-0.8809 (9)	-0.0539 (4)	5.71 (6)
C22	0.3034 (13)	-0.7501 (8)	-0.4718 (3)	6.90 (7)
C23	-0.4621 (11)	-0.5982 (6)	-0.0866 (3)	4.18 (4)
C24	-0.1461 (12)	-0.5388 (6)	-0.0977 (4)	3.79 (4)
C25	-0.3664 (12)	-0.5393 (6)	-0.1844 (3)	4.04 (4)

Table 2. Bond distances (Å) and angles ( $^\circ$ )

C8—Cr	2.278 (7)	C3—C2	1.503 (11)
C9—Cr	2.312 (6)	C4—C3	1.536 (10)
C11—Cr	2.256 (7)	C5—C4	1.562 (8)
C12—Cr	2.302 (6)	C18—C4	1.533 (9)
C13—Cr	2.233 (7)	C19—C4	1.525 (10)
C14—Cr	2.186 (8)	C6—C5	1.539 (10)
C23—Cr	1.773 (8)	C10—C5	1.543 (9)
C24—Cr	1.829 (9)	C7—C6	1.515 (10)
C25—Cr	1.838 (8)	C8—C7	1.490 (10)
C23—S	1.573 (8)	C9—C8	1.408 (9)

C24—O1	1.160 (8)	C14—C8	1.399 (9)
C25—O2	1.160 (8)	C10—C9	1.548 (8)
C12—O3	1.356 (8)	C11—C9	1.416 (9)
C21—O3	1.420 (9)	C20—C10	1.528 (10)
C19—O4	1.406 (8)	C12—C11	1.411 (9)
C22—O4	1.410 (9)	C13—C12	1.397 (10)
C2—C1	1.523 (10)	C14—C13	1.379 (10)
C10—C1	1.531 (9)		
C24—Cr—C23	85.6 (3)	C14—C8—C9	117.8 (7)
C25—Cr—C23	85.7 (3)	C10—C9—C8	121.2 (6)
C25—Cr—C24	88.2 (3)	C11—C9—C8	118.9 (6)
C21—O3—C12	117.2 (6)	C11—C9—C10	119.6 (6)
C22—O4—C19	112.2 (6)	C5—C10—C1	109.6 (6)
C10—C1—C2	112.6 (6)	C9—C10—C1	111.4 (5)
C3—C2—C1	111.6 (7)	C9—C10—C5	108.3 (5)
C4—C3—C2	114.8 (6)	C20—C10—C1	108.6 (6)
C5—C4—C3	107.7 (5)	C20—C10—C5	114.8 (6)
C18—C4—C3	107.4 (6)	C20—C10—C9	103.9 (5)
C18—C4—C5	108.4 (5)	C12—C11—C9	121.3 (7)
C19—C4—C3	110.9 (6)	C11—C12—O3	115.4 (6)
C19—C4—C5	113.3 (6)	C13—C12—O3	125.7 (6)
C19—C4—C18	108.8 (6)	C13—C12—C11	118.8 (7)
C6—C5—C4	113.8 (6)	C14—C13—C12	118.9 (6)
C10—C5—C4	117.0 (5)	C13—C14—C8	123.8 (7)
C10—C5—C6	110.1 (5)	C4—C19—O4	108.5 (6)
C7—C6—C5	110.6 (6)	S—C23—Cr	176.0 (5)
C8—C7—C6	114.6 (6)	O1—C24—Cr	176.2 (8)
C9—C8—C7	122.6 (6)	O2—C25—Cr	176.1 (7)
C14—C8—C7	119.6 (7)		

A solution of hexacarbonylchromium(0) (6.21 mmol) and 12,19-dimethoxypodocarpa-8,11,13-triene (5.18 mmol) in dibutyl ether/THF (162 ml, 12:1) was heated under reflux for 24 h. Workup gave a mixture (7.3:1) of the  $\alpha$  and  $\beta$  diastereoisomers of tricarbononyl[(8,9,11,12,13,14- $\eta$ )-12,19-dimethoxypodocarpa-8,11,13-triene]chromium(0) (98%). A solution of this mixture (0.19 mmol) and *cis*-cyclooctene (7.68 mmol) in benzene (8 ml) was irradiated (366 nm) under nitrogen at room temperature for 25 min. A solution of  $\text{Ph}_3\text{P}$  (0.23 mmol) in  $\text{CS}_2$  (2 ml) was then added to the red solution and the mixture was left standing without irradiation overnight. Filtration through Celite followed by flash chromatography (silica gel, hexane/diethyl ether) gave what is now established as the  $\alpha$  diastereoisomer of dicarbonyl[(8,9,11,12,13,14- $\eta$ )-12,19-dimethoxypodocarpa-8,11,13-triene]thiocarbonylchromium(0) (63%) in the form of orange needles; m.p. 378–380 K.

The infrared spectrum showed strong absorptions at 1941 and 1889  $\text{cm}^{-1}$  (CO), and at 1194  $\text{cm}^{-1}$  (CS). The NMR spectra confirmed the presence of a single stereoisomer:  $\delta_{\text{H}}(\text{CDCl}_3)$  1.05, H(3 ax.); 1.08, H(18)<sub>3</sub>; 1.27, H(20)<sub>3</sub>; 1.25–1.32, H(1 ax.), H(5); 1.62–1.72, H(2 ax.), H(2 eq.), H(6 ax.); 1.83, H(3 eq.); 1.93–2.01, H(1 eq.), H(6 eq.); 2.61, H(7 ax.); 2.63, H(7 eq.); 3.24, H(19); 3.31, 19-OCH<sub>3</sub>; 3.41, H(19); 3.68, 12-OCH<sub>3</sub>; 5.28, H(13); 5.44, H(11); 5.60, H(14);  $\delta_{\text{C}}$  18.3, C(2); 18.7, C(6); 27.0, C(20); 27.3, C(7); 27.5, C(18); 35.5, C(3); 36.6, C(1); 37.4, C(10); 37.8, C(4); 47.7, C(5); 56.0, 12-OCH<sub>3</sub>; 59.3, 19-OCH<sub>3</sub>; 76.1, C(19); 81.6, C(11); 81.8, C(13); 99.3, C(14); 105.9, C(8); 131.8, C(9); 143.5, C(12); 231.8, 231.9, (CO); 345.9, (CS). *m/z* 440 (*M*, 14), 384 (*M*–2CO, 100), 340 (*M*–2CO–CS, 63), 288 [*M*–Cr(CO)<sub>2</sub>CS, 80].

It was expected that the stereochemistry of the single monothiocarbonyl isomer isolated from the ligand-exchange reaction would reflect the stereochemistry of the major  $\alpha$  isomer present in the precursor mixture of  $\text{Cr}(\text{CO})_3$  complexes, so confirmation was sought by determination of the crystal structure.

An analogous refinement with inverted coordinates refined to  $R = 0.0471$ . Therefore the published atomic positions represent the true absolute structure of the molecule.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71286 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1032]

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## Structure of Ethylenediammonium catena-Poly[tris(2-pyridinecarboxylato- $\kappa\text{N},\kappa\text{O}$ )holmite- $\mu$ -2-pyridinecarboxylato- $\kappa\text{N},\kappa\text{O}:\kappa\text{O}$ ] Hexahydrate

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## Abstract

The structure consists of polymeric complex anions, ethylenediammonium cations and waters of crystallization. The Ho atoms are surrounded by four N atoms and five carboxylic O atoms. The compound is isomorphous with a previously published neodymium complex.

## Comment

The title compound is isomorphous with a neodymium compound, whose structure has been published previously (Starynowicz, 1991). This work was undertaken to obtain precise information about bond lengths within the holmium coordination sphere and their variation with a change of lanthanide cation.