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Structure and Absolute Configuration of the $(-)_D$ Isomer of Lithium Ethylenediamine-*N*,*N*′-diacetato-*N*,*N*′-di-3-propionatochromate(III) Pentahydrate, $(-)_D$-Li[Cr(EDDDA)]·5H_2O

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Assignment of the Λ absolute configuration to the complex anion $(-)_D$-[Cr(EDDDA)]$^-$, where EDDDA is the anion *N*,*N*′-diacetato-*N*,*N*′-di-3-propionatoethylenediamine, has been confirmed by a single-crystal x-ray diffraction study. The lithium salt crystallizes as red-violet octahedra in the orthorhombic space group $P2_12_12_1$ with $a = 11.635$ (3) Å, $b = 13.309$ (2) Å, $c = 12.633$ (2) Å, $V = 1956.2$ (7) Å^3, and $Z = 4$. Counter techniques were used to collect 1241 reflections of which 1139 had intensities greater than $3\sigma(I)$ where $\sigma(I)$ was determined from counting statistics. The structure was solved by Patterson, Fourier and least-squares techniques, and the model was refined isotropically to $R = 0.079$ and $R_w = 0.092$. The anomalous dispersion technique was used to assign the absolute configuration. The asymmetric unit contains a central chromium(III) ion coordinated octahedrally by a sexadentate EDDDA ligand, a lithium counterion coordinated tetrahedrally by an acetato oxygen atom and three oxygen atoms originating from water molecules, and two lattice waters of crystallization. A bridging acetato group joins each lithium ion to a chromium complex anion. Coordination of the chromium ion by EDDDA creates three five-membered rings and two six-membered rings. The five-membered acetate rings occupy trans-axial coordination sites while the two six-membered propionate rings lie in the same plane as the two donor nitrogen atoms (the girdle plane). Some pertinent interatomic distances and angles are Cr–O = 1.952 (10)–1.971 (10) Å (average 1.961 ± 0.009 Å), Cr–N = 2.072 (12) and 2.097 (12) Å, and Li–O = 1.947 (30)–1.983 (29) Å (average 1.959 ± 0.017 Å). Extensive hydrogen bonding involving both coordinated and uncoordinated carboxyl oxygen atoms of EDDDA, three water molecules coordinated to the lithium ion, and two lattice waters creates a rigid framework of structural moieties. These interactions may account for the formation of good-quality crystals with the lithium ion while other counterions yield salts of poor crystal quality.

Introduction

General methods for relating the absolute configuration of optically active metal chelates[1-3] to the signs of their CD curves, observed at wavelengths corresponding to visible absorption bands, are of continuing interest to inorganic chemists. In order to clarify the effects of chelate ring size and their arrangement around the central metal ion on the CD spectrum of optically active complexes, previous workers have prepared and characterized cobalt(III) complexes containing the anions of ethylenediamine-*N*,*N*′-diacetic-*N*,*N*′-3-propionic acid (H_4EDDDA),[4,5] ethylenediaminediacetic-*N*,*N*′-di-2-propionic acid (H_4EDPA),[6] and ethylenediamine-*N*,*N*′-disuccinic acid (H_4EDDS).[7] All of these complexes can be prepared under conditions which result in stereospecific chelation by the ligand. Stereospecificity in these cases produces geometrical isomers that have two six-membered rings in the equatorial plane of an octahedrally coordinated cobalt(III) ion. X-ray studies of the structurally related [Co(EDTA)]$^-$ complex have shown that the two glycinate rings, which lie in the plane of the two coordinated nitrogen atoms (G rings), are more strained than the two glycinate rings lying outside this plane (R rings). Presumably, there would be more strain in the G plane for five-membered rings than for six-membered rings, and the EDDDA, EDPA, and EDDS ligands would form preferentially two six-membered rings in the G plane.

In the case of H_4EDDDA, three geometric isomers which differ in the number (2, 1, or 0) of six-membered rings lying in the girdle plane are possible (Figure 1). With cobalt(III) both geometric isomers trans(O_5) and trans(O_5O_6) were prepared and characterized and the optical isomers were resolved.[4,5] The absolute configurations of these cobalt(III) complexes were assigned on the basis of the sign of their lowest energy CD peaks and their similarity with the CD spectrum of [Co(EDTA)]$^-$, which is of known absolute configuration.[2,8,9]

Table I. Crystal Data for LiCrC$_{12}$H$_{16}$N$_2$O$_8$·5H_2O

Fw 465.28
$a = 11.635$ (3) Å
$b = 13.309$ (2) Å
$c = 12.633$ (2) Å
$V = 1956.2$ (7) Å^3
$Z = 4$
$d_{\text{calcd}} = 1.57$ g cm^{-3}
$d_{\text{measd}}{}^a = 1.58$ (1) g cm^{-3}
$F(000) = 972$
μ(Cu Kα) = 56.69 cm^{-1}
Space group $P2_12_12_1$ from systematic absences: $h00$, $h \neq 2n$; $0k0$, $k \neq 2n$; $00l$, $l \neq 2n$

[a] Flotation: methylene chloride–bromoform.

With chromium(III) only one geometrical isomer, the (O_5), has been prepared with the ligands EDDDA and EDDS.[10] It is postulated that the larger Cr(III) ion induces more strain into the G plane for five-membered rings, and therefore, the trans(O_5) isomer is formed exclusively. Resolution of the optical isomers of [Cr(EDDDA)]$^-$ has been achieved and tentative assignment of the Λ configuration has been made for the $(-)_D$ isomer on the basis of a positive CD peak for the lowest energy d–d absorption band. In order to confirm this assignment of configuration, we report the results of an x-ray structure study on the lithium salt of the $(-)_D$ isomer, $(-)_D$-Li[Cr(EDDDA)]·5H_2O.

Experimental Section

Red-violet crystals of $(-)_D$-Li[Cr(EDDDA)]·5H_2O were prepared by the procedure described in ref 10. To minimize absorption effects several large, octahedrally shaped crystals were placed in a sphere grinder. Unfortunately, only cylindrical specimens were obtained and one of dimensions 0.33 × 0.33 × 0.42 mm was chosen for data collection. The crystal was found to be orthorhombic and room-temperature cell dimensions were determined by a least-squares fit of 15 reflections. Crystal data are shown in Table I.

Intensity data with $6.0 \leq 2\theta \leq 100.0°$ were collected on a Syntex $P2_1$ diffractometer in the θ–2θ scanning mode using graphite-monochromated Cu Kα radiation (λ 1.541 78 Å) and a variable scan speed. Periodically monitored check reflections showed no significant crystal deterioration. Of the 1241 independent reflections measured, 1139 had intensities greater than $3\sigma(I)$ where the net intensity I and the estimated standard deviation were calculated from $I = S(P - B_1 - B_2)$; $\sigma^2(I) = S(P + B_1 + B_2)$. Lorentz, polarization, and absorption corrections were applied. Transmission factors (0.048 < TF < 0.187) were calculated using a version of Busing's ORABS program modified for the orientation and rotation conventions of the Syntex $P2_1$ diffractometer.

Structure Determination and Refinement

From a three-dimensional Patterson calculation, a set of chromium atom coordinates was chosen. Successive structure factor calculations

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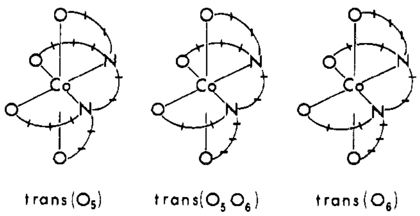

Figure 1. Geometric isomers of the ethylenediamine-*N*,*N'*-diacetato-*N*,*N'*-di-3-propionatocobalt(III) ion, [Co(EDDDA)]$^-$.

Table II. Positional (10^4) and Thermal Parameters for Atoms in the Structure of Λ(−)$_D$-Li[Cr(EDDDA)]·5H_2O

Atom	*x*/*a*	*y*/*b*	*z*/*c*	*B*, Å^2
Cr	−1475 (2)	1281 (2)	697 (2)	*a*
Li	−1800 (24)	2212 (20)	−3817 (22)	2.8 (5)
O(1)	−2129 (8)	1383 (7)	−726 (8)	2.1 (2)
O(2)	−2306 (9)	1814 (8)	−2409 (9)	3.2 (2)
O(3)	−2938 (9)	816 (8)	1313 (8)	2.5 (2)
O(4)	−4400 (10)	905 (9)	2405 (9)	3.5 (3)
O(5)	−926 (8)	−85 (7)	449 (7)	2.1 (2)
O(6)	38 (10)	−1373 (8)	1141 (9)	3.6 (2)
O(7)	−1830 (8)	2693 (7)	1004 (7)	2.0 (2)
O(8)	−1333 (9)	4276 (8)	733 (9)	3.5 (2)
O(9)	−751 (10)	3350 (8)	−3979 (9)	3.5 (2)
O(10)	−1038 (9)	992 (8)	−4380 (9)	3.3 (2)
O(11)	−3180 (10)	2611 (9)	−4586 (9)	3.8 (3)
O(12)	1350 (10)	713 (9)	−4203 (10)	4.4 (3)
O(13)	−2177 (11)	4923 (10)	−2713 (10)	4.7 (3)
N(1)	72 (10)	1921 (8)	176 (9)	1.7 (2)
N(2)	−672 (10)	1093 (9)	2148 (9)	2.2 (3)
C(1)	−1692 (13)	1662 (11)	−1608 (12)	2.3 (3)
C(2)	−378 (15)	1819 (13)	−1755 (14)	3.2 (4)
C(3)	398 (13)	1480 (11)	−870 (12)	2.3 (3)
C(4)	964 (14)	1555 (12)	987 (13)	3.0 (3)
C(5)	447 (14)	1692 (13)	2067 (13)	2.8 (3)
C(6)	−1361 (14)	1532 (11)	3061 (13)	2.8 (3)
C(7)	−2556 (14)	1029 (12)	3205 (13)	2.9 (3)
C(8)	−3352 (15)	949 (12)	2258 (13)	2.6 (3)
C(9)	−432 (13)	−526 (11)	1208 (11)	1.8 (3)
C(10)	−437 (13)	1 (12)	2287 (12)	2.5 (3)
C(11)	−1151 (13)	3352 (11)	646 (13)	2.4 (3)
C(12)	0 (14)	3000 (11)	181 (12)	2.5 (3)
H(1)	−120	1493	−2448	3.4
H(2)	−208	2544	−1934	3.4
H(3)	1226	1654	−1041	2.4
H(4)	393	726	−827	2.4
H(5)	1698	1961	934	3.0
H(6)	1141	819	880	3.0
H(7)	1011	1451	2635	2.9
H(8)	303	2426	2201	2.9
H(9)	−891	1471	3730	2.8
H(10)	−1443	2271	2952	2.8
H(11)	−2459	350	3543	2.9
H(12)	−2972	1369	3811	2.9
H(13)	337	−89	2656	2.4
H(14)	−1044	−306	2767	2.4
H(15)	661	3290	603	2.6
H(16)	84	3259	−570	2.6

[a] The anisotropic thermal parameters, defined by the expression $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$, refined to the values $\beta_{11} = 0.0020$ (2), $\beta_{22} = 0.0038$ (2), $\beta_{33} = 0.0018$ (2), $\beta_{12} = -0.001$ (1), $\beta_{13} = -0.0001$ (2) and $\beta_{23} = 0.0000$ (1).

Table III. Selected Bond Distances (Å) and Angles (deg) for the Structure of Λ(−)$_D$-Li[Cr(EDDDA)]·5H_2O

	Distances about Cr(III)		
Cr–O(1)	1.957 (10)	Cr–O(5)	1.952 (10)
Cr–O(3)	1.971 (10)	Cr–N(1)	2.097 (12)
Cr–O(7)	1.962 (10)	Cr–N(2)	2.072 (12)
	Distances about Li(I)		
Li–O(2)	1.947 (30)	Li–O(10)	1.983 (29)
Li–O(9)	1.955 (29)	Li–O(11)	1.949 (30)
	Intraligand Distances		
O(1)–C(1)	1.280 (18)	C(8)–O(4)	1.235 (20)
C(1)–O(2)	1.255 (19)	C(8)–O(3)	1.299 (19)
C(1)–C(2)	1.554 (23)	O(5)–C(9)	1.263 (17)
C(2)–C(3)	1.506 (23)	C(9)–O(6)	1.255 (18)
C(3)–N(1)	1.495 (19)	C(9)–C(10)	1.534 (21)
N(1)–C(4)	1.538 (20)	C(10)–N(2)	1.489 (20)
C(4)–C(5)	1.502 (23)	O(7)–C(11)	1.264 (18)
C(5)–N(2)	1.530 (20)	C(11)–O(8)	1.253 (19)
N(2)–C(6)	1.521 (20)	C(11)–C(12)	1.535 (22)
C(6)–C(7)	1.553 (23)	C(12)–N(1)	1.438 (18)
C(7)–C(8)	1.517 (23)		
	Intraligand Angles		
O(1)CrO(3)	92.8 (4)	C(5)N(2)C(10)	111.1 (12)
O(1)CrO(7)	91.9 (4)	C(6)C(7)C(8)	119.0 (14)
O(1)CrN(1)	91.0 (4)	C(7)C(8)O(4)	119.2 (14)
O(1)CrO(5)	92.5 (4)	C(7)C(8)O(3)	120.5 (14)
N(2)CrO(3)	90.1 (4)	O(4)C(8)O(3)	119.9 (14)
N(2)CrO(7)	92.1 (4)	C(8)O(3)Cr	129.8 (11)
N(2)CrN(1)	86.6 (5)	CrO(5)C(9)	117.4 (9)
N(2)CrO(5)	83.2 (4)	O(5)C(9)C(10)	117.4 (12)
O(3)CrO(5)	93.0 (4)	O(5)C(9)O(6)	124.4 (13)
O(3)CrO(7)	92.3 (4)	O(6)C(9)C(10)	118.2 (13)
O(7)CrN(1)	81.6 (4)	C(9)C(10)N(2)	110.0 (12)
N(1)CrO(5)	92.7 (4)	CrO(7)C(11)	117.5 (10)
CrO(1)C(1)	131.7 (10)	O(7)C(11)O(8)	122.9 (13)
O(1)C(1)O(2)	121.4 (13)	O(7)C(11)C(12)	118.0 (13)
O(1)C(1)C(3)	122.3 (13)	O(8)C(11)C(12)	118.7 (13)
O(2)C(1)C(2)	116.2 (13)	C(11)C(12)N(1)	110.9 (12)
C(1)C(2)C(3)	117.4 (14)	C(1)O(2)Li	127.5 (15)
C(2)C(3)N(1)	112.7 (12)	O(2)LiO(9)	119.7 (14)
C(3)N(1)Cr	109.6 (8)	O(2)LiO(10)	103.9 (13)
C(3)N(1)C(12)	114.3 (11)	O(2)LiO(11)	106.3 (14)
C(3)N(1)C(4)	107.1 (11)	O(9)LiO(10)	108.5 (13)
CrN(1)C(4)	103.9 (8)	O(10)LiO(11)	114.4 (13)
CrN(1)C(12)	110.7 (9)	O(9)LiO(11)	104.6 (13)
C(12)N(1)C(4)	110.7 (11)	N(2)C(6)C(7)	113.2 (12)
N(1)C(4)C(5)	107.3 (13)	CrN(2)C(10)	107.8 (11)
C(4)C(5)N(2)	109.8 (13)	CrN(2)C(6)	112.8 (11)
C(5)N(2)C(6)	107.4 (11)	C(6)N(2)C(10)	112.4 (11)
C(5)N(2)Cr	105.2 (12)	C(5)N(2)Cr	105.2 (12)

and electron density maps revealed the positions of all remaining nonhydrogen atoms. Both block-diagonal and full-matrix least-squares calculations were employed to refine the model. Anisotropic temperature factors were assigned to the chromium atom but isotropic thermal parameters were used for all other atoms. Refinement of the model lowered the residuals to $R = 0.084$ and $R_w = 0.098$. Least-squares refinement using coordinates for the alternate enantiomorph converged to $R = 0.120$ and $R_w = 0.132$. Comparison of Friedel pairs also confirmed the assignment.

Positions for 16 hydrogen atoms were calculated from the heavier atom coordinates. The hydrogen atom contributions to the structure factors were included in a final block-diagonal least-squares calculation, but the coordinates were not refined. Isotropic temperature factors for the hydrogen atoms were fixed at the value of the temperature factor of the associated heavier atom. With 29 nonhydrogen atoms in the asymmetric unit, 122 variable parameters, and 1139 observables, the final discrepancy factors were $R = 0.079$ and $R_w = 0.092$. Positional and thermal parameters from the last cycle of least-squares refinement are found in Table II; a table of observed and calculated structure factors is available as supplementary material.

The analytical scattering factors of Cromer and Waber[11] were used for neutral lithium, chromium, carbon, nitrogen, and oxygen atoms, and corrections for the real and imaginary components of the anomalous dispersion were applied to the chromium scattering factors. The scattering factors for hydrogen atoms were those of Stewart, Davidson, and Simpson.[12] The function minimized during the least-squares refinement was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma^2$ and $\sigma^2 = \sigma^2(I) + (0.01I)^2$. The discrepancy factors are defined by

$$R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$$

$$R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$$

All calculations were carried out on a Xerox Sigma 9 computer system. In addition to locally written programs, Zalkin's FORDAP Fourier summation program, Okaya's block-diagonal least-squares and distance–angle program, Ibers' NUCLS modification of the Busing–Martin–Levy ORFLS full-matrix least-squares program, and Johnson's

Table IV. Oxygen–Oxygen Distances (Å) Indicating Possible Hydrogen Bonds in the Structure of (–)$_D$-Li[Cr(EDDDA)]·5H$_2$O

O(2)–O(10)	3.09 (2)	O(6)–O(11)[a]	2.87 (2)	O(9)–O(13)	3.11 (2)
O(2)–O(11)	3.12 (2)	O(8)–O(12)[d]	2.72 (2)	O(10)–O(12)	2.81 (2)
O(3)–O(10)[a]	2.83 (1)	O(8)–O(13)[e]	2.83 (2)	O(11)–O(12)[f]	2.76 (2)
O(4)–O(9)[b]	2.72 (2)	O(9)–O(10)	3.20 (2)	O(12)–O(13)[c]	2.81 (2)
O(6)–O(9)[c]	2.88 (2)	O(9)–O(11)	3.09 (2)		

[a] $-{}^1/_2 - x, -y, {}^1/_2 + z$. [b] $-{}^1/_2 + x, -y, {}^1/_2 + z$. [c] $-x, -{}^1/_2 + y, -{}^1/_2 - z$. [d] $-x, {}^1/_2 + y, -{}^1/_2 - z$. [e] $-{}^1/_2 - x, 1 - y, {}^1/_2 + z$. [f] $-{}^1/_2 + x, {}^1/_2 - y, -1 - z$.

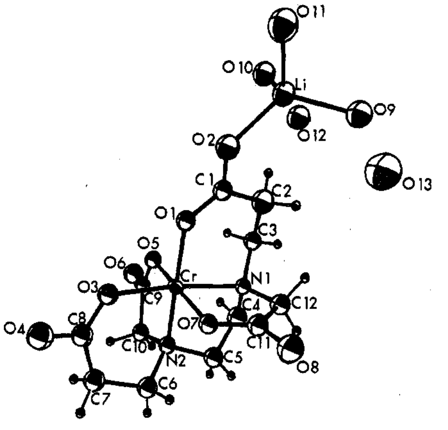

Figure 2. Asymmetric structural unit for (–)$_D$-Li[Cr(EDDDA)]·5H$_2$O.

ORTEP plotting program were employed.

Discussion

Figure 2 shows the asymmetric structural unit and the atomic numbering scheme used in all tables and figures. Selected bond distances and angles are presented in Table III.

The chromium(III) ion is coordinated octahedrally by the sexadentate ligand, EDDDA. Four oxygen atoms, O(1), O(3), O(5), and O(7), from carboxyl groups are statistically equidistant from the Cr(III) ion with an average distance of 1.961 ± 0.0009 Å. The two nitrogen atoms N(1) and N(2) complete the octahedral coordination at distances of 2.097 (12) and 2.072 (12) Å, respectively. The arrangement of these donor atoms creates three five-membered and two six-membered chelate rings. Since two five-membered acetate rings are trans to each other, the three remaining chelate rings are in the equatorial plane and three skew-chelate ring pairs result. By the IUPAC conventions,[13] the system would be designated as containing "skew-chelate pairs, ΛΔΛ", and the overall configuration of the sexadentate complex is Λ. This designation is the same as that assigned by the CD technique[10] and the identification of the trans(O$_5$) isomer via infrared arguments is confirmed.

The lithium counterion is coordinated tetrahedrally by four oxygen atoms. The O(2) atom originates from a carboxyl group which serves to bridge the Cr(III) and Li ions. The O(9), O(10), and O(11) atoms are from water molecules which are hydrogen-bonded to other structural entities. The Li–O

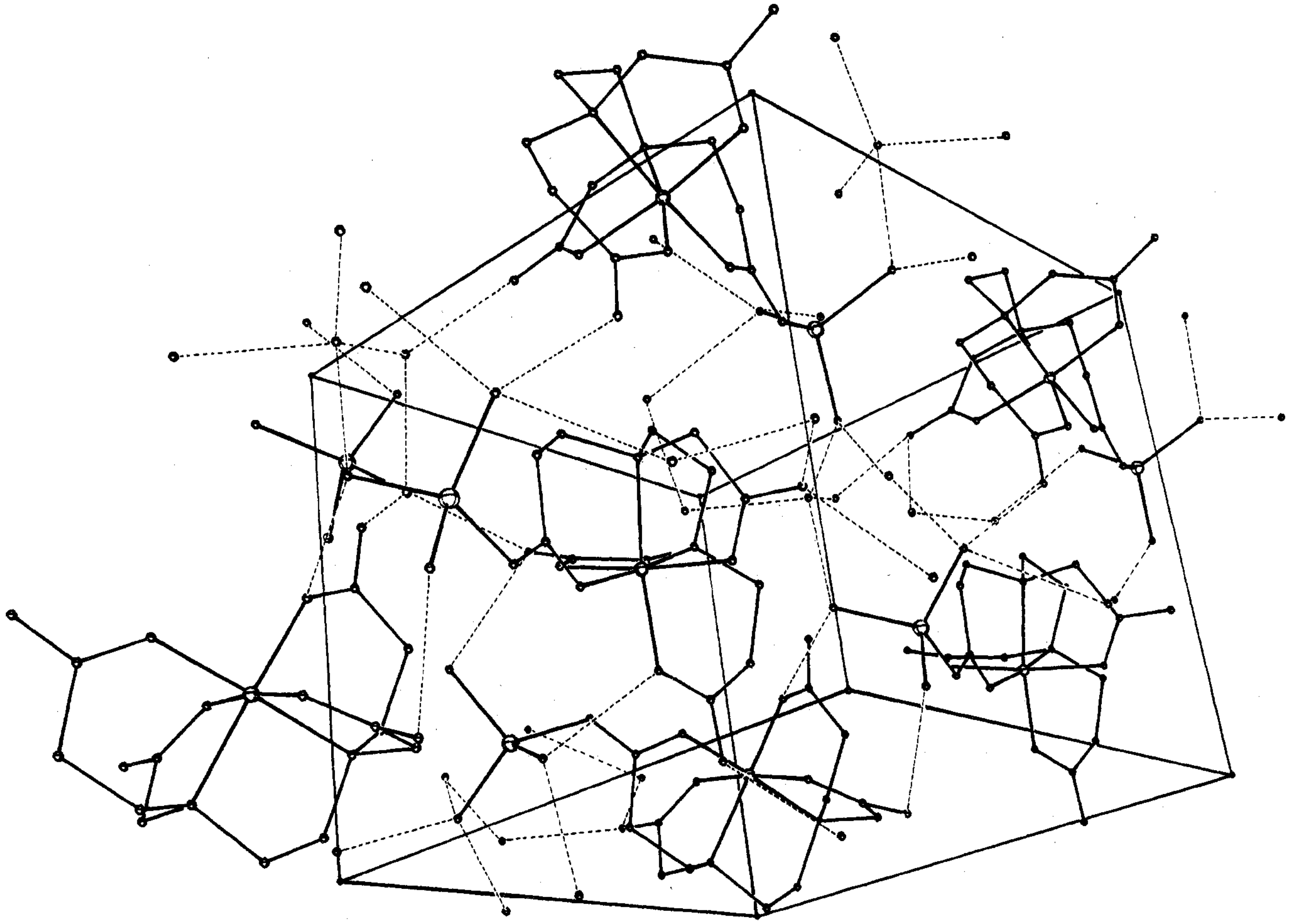

Figure 3. Packing diagram showing hydrogen bonding network for (–)$_D$-Li[Cr(EDDDA)]·5H$_2$O.

distances are well within the accepted range of values with an average of 1.959 ± 0.017 Å.

The extensive hydrogen-bonded network is shown in the packing diagram, Figure 3, and important distances are listed in Table IV. While three of the coordinating carboxyl oxygen atoms, O(1), O(5), and O(7), are not involved in any significant hydrogen bonding (i.e., O···O > 3.2 Å), all other carboxyl oxygen atoms participate in the formation of hydrogen bonds. The two water molecules of crystallization, represented by oxygen atoms O(12) and O(13), also serve to extend the network; in fact, O(12) is involved in a total of four hydrogen bonds. The three water molecules bound to the lithium ion, O(9), O(10), and O(11), play an important role in binding the structural units together. Each of these water molecules is hydrogen bonded to a carboxyl oxygen atom on an adjacent $[Cr(EDDDA)]^-$ anion. This extensive three-dimensional network may account for good-quality crystals produced with the lithium counterion. Counterions such as K^+ do not yield useful crystals in the purification process.[4,10] The Li(I) ion may be of optimum size for the development of the strongly interacting three-dimensional structure.

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Registry No. $(-)_D$-Li[Cr(EDDDA]·$5H_2O$, 63301-38-2.

Supplementary Material Available: Listing of structure factor amplitudes (6 pages). Ordering information is given on any current masthead page.

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Contribution from the Wright and Rieman Chemistry Laboratories, Rutgers, The State University of New Jersey, New Brunswick, New Jersey 08903, and The Weizmann Institute of Science, Rehovot, Israel

Molecular Structure of μ-Diphenylacetylene-[1-{carbonyl(η^4-tetraphenylcyclobutadiene)molybdenum(0)}-2-{dicarbonyl-μ-(η^4-tetraphenylcyclopentadienone)-molybdenum(0)}], $(OC)_2(Ph_4C_5O)Mo(Ph_2C_2)Mo(C_4Ph_4)(CO)$, a Complex with a Mo═Mo Bond and a Bridging Cyclopentadienone Group

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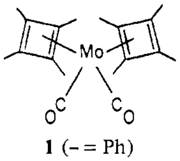
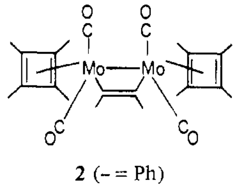
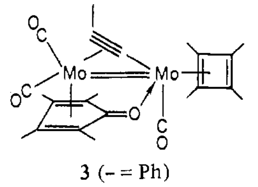
The crystal structure of the title compound, formed from the reaction of $Mo(CO)_6$ and Ph_2C_2 at elevated temperature and pressure, has been determined by a single-crystal x-ray diffraction study. The structure consists of binuclear molybdenum units bonded to three terminal carbonyls and one diphenylacetylene, one tetraphenylcyclobutadiene, and one tetraphenylcyclopentadienone ligand. The Mo–Mo distance [2.772 (4) Å] is consistent with the assignment of a Mo–Mo double bond. Diphenylacetylene acts as a bridging ligand with the C–C bond almost perpendicular (83°) to the Mo–Mo bond axis, while the tetraphenylcyclobutadiene group bonds axially to one of the molybdenum atoms; neither of these modes of attachment is unusual. An exceptional feature of the structure is the attachment of the tetraphenylcyclopentadienone ligand: all five cyclopentadienone carbon atoms are bonded to Mo(2) [Mo(2)–C = 2.30 (2)–2.39 (2) Å] while the oxygen atom is bonded to Mo(1) [Mo(1)–C = 2.06 (1) Å], demonstrating that the ligand is bridging. Crystallographic data are as follows: space group $P2_1/a$; $a = 23.96$ (2), $b = 22.14$ (3), $c = 12.49$ (2) Å; $\beta = 110.24$ (2)°; $Z = 4$. The structure was solved by a combination of heavy-atom and direct methods and refined to a conventional R value of 0.111 for 4811 independent reflections.

Introduction

In 1964, Hübel and Merênyi[2a] reported the formation of several molybdenum complexes from the reaction of diphenylacetylene with either $Mo(CO)_6$ or $(diglyme)Mo(CO)_3$. Particularly noteworthy are the complexes with assigned structures **1** and **2**. The structure of **1**, the only known complex with a dicyclobutadiene geometry, has been confirmed recently by a single-crystal x-ray analysis.[2b] The structure originally proposed for the green, diamagnetic binuclear complex **2** suggests the presence of metal atoms with 16-electron configurations and is further characterized by an unusual bridging geometry for the diphenylacetylene ligand. Here, we present a crystallographic study of this complex, detailing its structure as **3** rather than **2**.


1 (– = Ph)


2 (– = Ph)


3 (– = Ph)

Experimental Section

Synthesis. A sample of the title compound was prepared by a modification of the Hübel and Merênyi procedure. A mixture consisting of $Mo(CO)_6$ (10.8 g, 41 mmol) and Ph_2C_2 (10.8 g, 61 mmol) in 90 mL of benzene was charged into a 300 mL autoclave and heated with stirring to 120 (1) °C for 17 h. The product mixture was filtered, and the filtrate was concentrated to dryness under reduced pressure (~1 mm; 30 °C). Unreacted $Mo(CO)_6$ was removed by sublimation (0.05 mm; 80 °C). The residue was dissolved in benzene–petroleum ether (bp 30–60 °C) (1/10, v/v), and the resulting solution was