which Mo—N *trans* to nitrogen is shorter [2·149 (6) Å], consistent with the change in formal oxidation state, and Mo—N *trans* to the stronger π-acceptor CO ligand is longer [2·230 (6) Å] (Chisholm, Connor, Huffman, Kober & Overton, 1984), and *fac*-$[Mo(CO)_3(bpy)(py)]$ in which Mo—N(bpy) is 2·235 (7) Å (Griffiths, 1971).

The Mo—C distances are of two types: (*a*) C(1*b*) *trans* to nitrogen, N(12), 2·096 (14) Å, which compares with the shortest Mo—C distance in $[Mo(dppm)(CNMe)_5]^{2+}$ [2·090 (9) Å] and in $[Mo(dppe)(CNMe)_5]^{2+}$ [2·096 (7) Å] (Dewan, Wood, Walton & Lippard, 1982);* and (*b*) C(1*a*), C(1*c*) (ave. 2·028 Å) which are among the shortest measured for molybdenum(II)–isocyanide complexes, which typically contain Mo—CN*R* bond lengths in the range 2·04—2·16 Å. The dimensions of the isocyanide ligands in $[Mo(bpy)_2(CNEt)_3]^{2+}$ show two features: (*a*) the C≡N bond is slightly longer (ave. 1·164 Å) than in other molybdenum(II)–isocyanide complexes {*e.g.* 1·14 Å in $[Mo(CNEt)_7]^{2+}$ (Brant, Cotton, Sekutowski, Wood & Walton, 1979), 1·144 Å in $[Mo(dppe)(CNMe)_5]^{2+}$ (Dewan *et al.*, 1982)} consistent with the greater σ-donor strength of two bpy ligands, and (*b*) the CNC angle (ave. 161·0°) is significantly distorted in all three isocyanide ligands from its typical value (177°) in similar structures (Brant *et al.*, 1979; Dewan *et al.*, 1982).

Oxidation has no significant influence on the bpy ligand bite angle {$[Mo(bpy)_2(CNEt)_3]^{2+}$ (ave. 72·7°); *cis*-$[Mo(CO)_2(bpy)_2]$ (ave. 73·6°) (Chisholm *et al.*, 1984)}. Based on bpy ligand dimensions, the average charge on the bpy ligand in $[Mo(bpy)_2(CNEt)_3]^{2+}$ is similar to that in *cis*-$[Mo(CO)_2(bpy)_2]$ (Chisholm *et al.*, 1984) and less than that in $[Mo(OPr^i)_2(bpy)_2]$ (Chisholm, Huffman, Rothwell, Bradley, Kress & Woodruff, 1981).

The absence of both a mirror plane in the cation and a twofold axis passing through Mo—C(1*b*)—N(1*b*), together with the facts that (*a*) the Mo atom is not coplanar with the isocyanide donor atoms C(1*a*), C(1*b*), and C(1*c*), (*b*) there is a large dihedral angle between the planes of the bpy ligands and (*c*) the value of the normalized bite (Kepert, 1979) of the bpy ligand is small ($b = 1·18$), all indicate the low symmetry of the cation. The isocyanide-capped distorted octahedral geometry at molybdenum in this salt is a further example of stereochemistry V (Kepert, 1979) in the $[M(\text{bidentate})_2(\text{unidentate})_3]^{x\pm}$ series.

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* dppm is bis(diphenylphosphino)methane, dppe is 1,2-bis(diphenylphosphino)ethane.

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catena-Tri-μ-chloro-bis(2,6-dimethyl-4-pyrone)lanthanum(III), $[LaCl_3(C_7H_8O_2)_2]_n$

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Abstract. $M_r = 493·55$, monoclinic, $P2_1$ or disordered $P2_1/m$, $a = 11·202$ (7), $b = 7·944$ (1), $c = 10·011$ (6) Å, $\beta = 102·55$ (7)°, $V = 869·6$ (5) Å^3, $Z = 2$, $D_x = 1·885$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 29·7$ cm^{-1}, $F(000) = 480$, $T = 293$ K. Final $R = 3·5\%$ for 1697 unique observed reflections. Each La$^{\text{III}}$

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is coordinated by six chlorines, equivalent in pairs, and by the two non-equivalent $C_7H_8O_2$ carbonyl oxygens; the coordination polyhedron is a slightly distorted square antiprism. The coordination polyhedra form infinite polymeric chains running along **b**, by sharing three chlorines between each pair of nearest La atoms. La—O($C_7H_8O_2$): 2·433 (6) and 2·485 (5) Å; La—Cl: 2·845 (2)–3·018 (3) Å.

Introduction. Lanthanoid trichlorides form a series of complexes by coordinating 2,6-dimethyl-4-pyrone (dmp) with formula [$LnCl_3(dmp)_3$] (Castellani Bisi, 1970) where Ln represents all lanthanoids except promethium. The crystal structure of [$GdCl_3(dmp)_3$], typical of the series, was determined (Bisi Castellani & Tazzoli, 1984*a*), and was found to consist of monomeric units, with the metal ion in octahedral geometry.

Although the coordination number six is rather low for lanthanoids, especially for the lighter ones, in this series even lanthanum exhibits octahedral coordination; the crystal structure of [$LaCl_3(dmp)_3$] has not been determined; however, crystals of this complex are isomorphous with [$GdCl_3(dmp)_3$], as shown by its space group and lattice parameters determined in the course of this investigation: $P2_1/c$, $a = 10{\cdot}75$ (3), $b = 14{\cdot}92$ (4), $c = 18{\cdot}34$ (5) Å, $\beta = 120{\cdot}7$ (7)° compared with the corresponding figures for the Gd complex in the same space group [10·61 (1), 14·76 (1), 18·03 (1) Å, 120·0 (1)°].

In the case of lanthanum the complex [$LaCl_3(dmp)_2$] was also obtained; a polymeric structure involving chlorine bridges was assumed, as a monomeric form would imply five coordination, which is exceedingly rare for lanthanoids (Castellani Bisi, 1970). During the preliminary attempts to grow single crystals of this compound suitable for structure determination, a novel polymeric complex of formula [$LaCl_3(dmp)H_2O$]$_n$ was identified; it was formed by chance, presumably due to imperfect drying of the reaction medium. Its structure consists of infinite linear chains in which lanthanum is heptacoordinated, through the formation of chlorine bridges (Bisi Castellani & Tazzoli, 1984*b*). Recently crystals of [$LaCl_3(dmp)_2$]$_n$ suitable for X-ray diffraction were successfully grown, and the crystal structure determination is reported herein.

Experimental. Colourless prismatic fragment 0·017 × 0·017 × 0·033 cm; Philips PW1100 diffractometer with graphite monochromator; lattice-parameter measurements and refinement with a Philips routine (*LAT*) that takes into account a 2θ-circle zero-correction by measuring very accurate profiles in the positive and negative range of 2θ: 24 reflections in the range $15 \leq \theta \leq 16°$ used. 5090 reflections with $2 \leq \theta \leq 30°$ [$(\sin\theta)/\lambda \leq 0{\cdot}70$ Å^{-1}], $\pm h$, $\pm k$, l set with maximum absolute values 15, 11, 14 respectively; no intensity deterioration of standard reflections (400, 040, 004) measured every 4 h (the intensity variation was random and within 3% with respect to the mean), Lp correction, analytical absorption correction ($\mu_{\rm Mo} = 29{\cdot}7$ cm^{-1}) applied to the sphere of equal volume (diameter = 0·028 cm) plus North, Phillips & Mathews (1968) empirical correction by azimuthal scan of six reflections with $\chi = 86{\cdot}9°$ (120, 240, 360, $\bar{1}\bar{2}0$, $\bar{2}\bar{4}0$, $\bar{3}\bar{6}0$); range of the correction factors applied to the intensities: 0·993–1·143; averaging of reflections equivalent in pairs yielded 2716 independent observations, with merging factor $R_{\rm int} = 0{\cdot}054$ for the whole observed sphere and $R_{\rm int} = 0{\cdot}028$ for the 1697 observations with $I \geq 3\sigma(I)$; Patterson structure determination; full-matrix least-squares refinement with the F magnitudes by *ORFLS* (Busing, Martin & Levy, 1962); the 1019 F's with $I \leq 3\sigma(I)$ were not used in the refinement; weight = $1/\sigma^2(F)$, anisotropic thermal parameters for all atoms, H atoms not taken into consideration owing to disordering of dmp's; secondary-extinction coefficient according to Zachariasen (1963), refined to final $g = 1{\cdot}2\ (2) \times 10^{-5}$; atomic scattering factors with anomalous-dispersion coefficients from *International Tables for X-ray Crystallography* (1974); final $R = 0{\cdot}035$ for 1697 reflections ($R = 0{\cdot}070$ including all reflections), $R_w = 0{\cdot}0288$, goodness of fit $S = 1{\cdot}06$; $(\Delta/\sigma)_{\max} = 0{\cdot}07$, $\Delta\rho$ peaks in final difference map between $-0{\cdot}89$ and $+1{\cdot}17$ e Å^{-3}.

The crystal structure determination deserves additional comments; the La coordinates were deduced from the Patterson synthesis, and the La-phased Fourier map showed all chlorine positions, of course with $P2_1/m$ ambiguity; the correct chlorine sites in $P2_1$ were chosen by avoiding Cl—Cl contacts that were too close in the La coordination. It was quite surprising not to find the dmp molecules on the next Fourier map phased with La and three Cl's in $P2_1$. Instead, the dmp's could be found from the previous map, phased only with La. The $P2_1/m$ ambiguity applied to two different dmp's produced four different $P2_1$ hypotheses, which were refined isotropically (La only anisotropically) with final $R = 0{\cdot}068$, 0·066, 0·075 and 0·086 respectively; therefore the last two were rejected. The first was also rejected later because of unacceptably short Cl—dmp distances. The second model could not be refined further in $P2_1$. Only a disordered structure by 'mixing' the two $P2_1$ enantiomorphic images (sterically incompatible) could be refined in $P2_1/m$ by giving half weight to all atoms except La placed in the mirror plane. The refinement in $P2_1$ yielded the final $R_w = 0{\cdot}065$ and worse distances and angles than those found by allowing for disorder in $P2_1/m$, with final $R_w = 0{\cdot}029$. Disordered models in $P2_1$ – *i.e.* $P2_1$ Cl atoms with mirror half-images of dmp's, and *vice versa* – again could not be refined: final R_w's were 0·053 and 0·044 respectively and the distances and angles were worse than in statistical $P2_1/m$.

The structure disorder could fall halfway between random and domain disorder (Dunitz, 1979) as the intensity statistics – after a very good intensity scaling – give ambiguous figures halfway between centrosymmetric and non-centrosymmetric models (the only unambiguous figures refer correctly to the $h0l$ zone as centrosymmetric). The bond distances and angles of the dmp's are unsatisfactory and this very likely reflects the state of things (Brown, Norment & Levy, 1957). Further attempts to settle the question would require a specific theory and a very complex refinement program; this was judged to be not worth while and beyond the purposes of this investigation.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters,* B_{eq}, *with e.s.d.'s in parentheses*

$B_{\rm eq}$ according to Hamilton (1959), in Å^2. The site-occupation factors (in $P2_1/m$) are 0·5 for all atoms.

	x	y	z	$B_{\rm eq}$
La	4532 (1)	2500	5503 (1)	1·73 (1)
Cl(1)	5173 (2)	688 (3)	3172 (2)	2·21 (5)
Cl(2)	6308 (2)	−118 (3)	6690 (2)	2·67 (6)
Cl(3)	3229 (2)	−828 (3)	4883 (2)	2·48 (6)
O(1)	4213 (5)	1924 (6)	7840 (5)	2·86 (17)
O(2)	2391 (5)	2997 (7)	5512 (6)	3·21 (18)
O(3)	2656 (6)	−901 (7)	10349 (6)	2·19 (17)
O(4)	80 (6)	5671 (8)	7272 (6)	2·84 (18)
C(1)	3767 (8)	996 (12)	8627 (9)	2·82 (27)
C(2)	3794 (7)	−834 (10)	8653 (8)	1·81 (21)
C(3)	3195 (26)	−1652 (30)	9404 (26)	2·18 (41)
C(4)	3266 (29)	−3535 (26)	9704 (25)	2·60 (47)
C(5)	2627 (8)	819 (10)	10429 (8)	2·30 (24)
C(6)	1907 (8)	1478 (11)	11463 (9)	3·49 (29)
C(7)	3155 (7)	1807 (10)	9628 (8)	1·94 (20)
C(8)	1673 (8)	3836 (11)	6065 (9)	2·55 (25)
C(9)	1583 (8)	5698 (11)	5970 (9)	2·70 (24)
C(10)	772 (23)	6636 (27)	6409 (22)	2·44 (37)
C(11)	613 (24)	8509 (23)	6703 (23)	2·69 (45)
C(12)	129 (8)	3922 (13)	7430 (9)	3·00 (27)
C(13)	−725 (8)	3231 (11)	8275 (9)	3·63 (28)
C(14)	905 (7)	3044 (9)	6845 (8)	2·20 (21)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Key for symmetry transformations: (i) $1-x$, $\frac{1}{2}+y$, $1-z$; (ii) $1-x$, $-\frac{1}{2}+y$, $1-z$.

La–Cl(1)	2·960 (3)	La–Cl($1^{\rm i}$)	2·845 (2)
La–Cl(2)	2·944 (3)	La–Cl($2^{\rm i}$)	2·896 (3)
La–Cl(3)	3·018 (3)	La–Cl($3^{\rm i}$)	2·937 (3)
La–O(1)	2·485 (5)	La–O(2)	2·433 (6)
Cl(1)–La–Cl(2)	73·52 (7)	Cl(3)–La–Cl($2^{\rm i}$)	110·71 (7)
Cl(1)–La–Cl(3)	66·50 (6)	Cl(3)–La–Cl($3^{\rm i}$)	139·25 (5)
Cl(1)–La–Cl($1^{\rm i}$)	140·99 (5)	Cl(3)–La–O(1)	82·53 (13)
Cl(1)–La–Cl($2^{\rm i}$)	79·31 (7)	Cl(3)–La–O(2)	73·14 (13)
Cl(1)–La–Cl($3^{\rm i}$)	75·87 (7)	Cl($1^{\rm i}$)–La–Cl($2^{\rm i}$)	75·97 (7)
Cl(1)–La–O(1)	139·63 (12)	Cl($1^{\rm i}$)–La–Cl($3^{\rm i}$)	69·06 (6)
Cl(1)–La–O(2)	119·35 (14)	Cl($1^{\rm i}$)–La–O(1)	75·57 (12)
Cl(2)–La–Cl(3)	73·63 (7)	Cl($1^{\rm i}$)–La–O(2)	82·45 (13)
Cl(2)–La–Cl($1^{\rm i}$)	116·15 (7)	Cl($2^{\rm i}$)–La–Cl($3^{\rm i}$)	75·56 (8)
Cl(2)–La–Cl($2^{\rm i}$)	147·90 (3)	Cl($2^{\rm i}$)–La–O(1)	138·12 (13)
Cl(2)–La–Cl($3^{\rm i}$)	81·44 (7)	Cl($2^{\rm i}$)–La–O(2)	74·86 (14)
Cl(2)–La–O(1)	73·31 (13)	Cl($3^{\rm i}$)–La–O(1)	120·60 (14)
Cl(2)–La–O(2)	133·90 (13)	Cl($3^{\rm i}$)–La–O(2)	142·94 (13)
Cl(3)–La–Cl($1^{\rm i}$)	151·36 (5)	O(1)–La–O(2)	71·49 (18)
La–La$^{\rm ii}$	4·284 (2)	La–Cl(2)–La$^{\rm ii}$	94·36 (8)
La–Cl(1)–La$^{\rm ii}$	95·08 (6)	La–Cl(3)–La$^{\rm ii}$	91·99 (6)

The lattice parameters of $[\mathrm{LaCl_3(dmp)_3}]$ (see *Introduction*) were determined with a Philips diffractometer by scanning 22 reflections in the range $2 \le \theta \le 6°$, with $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}7107$ Å.

Discussion. Table 1 shows the atomic parameters, Table 2 the main bond distances and angles (they are quite unsatisfactory for the dmp's, for the reasons given above).* The structure (Fig. 1) is characterized by infinite polymeric chains running along **b**, formed by sharing three Cl's between each pair of nearest La atoms. The La coordination polyhedron is a slightly distorted square antiprism; the vertices of the quadrangular faces are occupied by three Cl atoms and the carbonylic O of one molecule of dmp [O(1), Cl(2), Cl($3^{\rm i}$), Cl($1^{\rm i}$) and O(2), Cl($2^{\rm i}$), Cl(1), Cl(3)].

This structure may be compared with that of $[\mathrm{LaCl_3(dmp)H_2O}]_n$ mentioned above. Both complexes achieve a coordination number higher than six, as expected for La, through Cl bridges; however, while bridges with two Cl atoms are quite common and a few examples of dimeric doubly bridged chloro-complexes of lanthanoids are known (Hodgson & Raymond, 1972; Atwood & Smith, 1973; Baker, Brown & Raymond, 1975), triple chlorine bridges are more unusual (Hodgson, 1975) and the structure described appears to be the first example reported for a lanthanoid complex. The bridges (Table 2) are definitely asymmetrical, as reported also for μ-dichloro-bis-[cyclotetraenylbis(tetrahydrofuran)cerium] (Hodgson & Raymond, 1972), whereas in $[\mathrm{LaCl_3(dmp)H_2O}]_n$, in

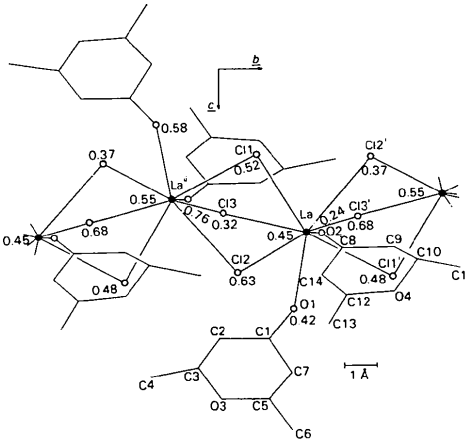

Fig. 1. View of the chain running along **b** with atom numbering. x coordinates are reported for the atoms of the La-coordination polyhedra.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39801 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

di-μ-chloro-bis[bis(η-cyclopentadienyl)scandium(III)] (Atwood & Smith, 1973) and in the ytterbium analogues (Baker, Brown & Raymond, 1975), they are symmetrical.

The asymmetry in the cerium complex was ascribed to the large radius of Ce, compared to that of Sc and Yb and to the corresponding increase in metal–metal distance, which does not allow the chloride ion to span the gap. However, the La—La^{ii} distance in the title compound [4·284 (2)] Å is shorter than the La—La distance in $[LaCl_3(dmp)H_2O]_n$, 4·73 (1) Å; this means that the asymmetry of chlorine bridges is not related to the intermetallic distance. The La—Cl bridging distances are longer on average (2·85–3·02 Å) than those found in $[LaCl_3(dmp)H_2O]_n$ (2·90–2·92 Å), reflecting the crowd of sharing chlorines around La in the title compound.

La—Cl—La angles [91·99 (6), 94·36 (8), 95·08 (6)°] fall in the range 70–100° like the majority of $M—X—M$ angles (Cotton & Wilkinson, 1980). The La—O distances [2·433 (6) and 2·485 (5) Å] are longer than the La—O(carbonyl) distance [2·33 (2) Å] in $[LaCl_3(dmp)H_2O]_n$, probably due to the more crowded environment about La in the title compound. In $[La(dmp)_8](ClO_4)_3$, which also exhibits a square-antiprism geometry, the average La—O bond length is 2·48 Å (Castellani Bisi, Della Giusta, Coda & Tazzoli, 1974).

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Chloro[1,1′-diphenyl-3,3′-(ethylenediimino)di-1-butanonato]iron(III), $[FeCl(C_{22}H_{22}N_2O_2)]$

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Abstract. $M_r = 437·74$, monoclinic, $P2_1/n$, $a = 12·554$ (3), $b = 15·832$ (4), $c = 10·250$ (3) Å, $\beta = 93·22$ (2)°, $V = 2034·02$ Å^3, $Z = 4$, $D_x = 1·429$ g cm^{-3}, $\lambda = 0·71069$ Å, $\mu = 8·32$ cm^{-1}, $F(000) = 908$, $T = 298$ K, final $R = 0·043$ for 2676 observed $[F_o > 3\sigma(F_o)]$ reflections. The Fe^{III} ion shows a distorted square-pyramidal geometry with the N_2O_2 atoms of the ligand at the base [Fe—N 2·051 (5) and 2·056 (5) Å; Fe—O 1·906 (4) and 1·925 (4) Å] and the Cl$^-$ at the apex [Fe—Cl 2·236 (3) Å].

Introduction. The tetradentate Schiff base *N,N′*-ethylenebis(benzoylacetonimine), H_2bzen, $C_{22}H_{24}N_2O_2$ (Haider, Hashem, Malik & Hursthouse, 1981), has been shown to form monomeric square-planar complexes with divalent Co (Haider, Hashem, Malik & Hursthouse, 1980), Ni (Malatesta & Mugnoli, 1981), Cu and Pd (Malik, Haider, Hashem & Hursthouse, 1985), all of which are isostructural. The structure of the corresponding Fe^{III} complex, $[FeCl(C_{22}H_{22}N_2O_2)]$, is now described.

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