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$_2$bzen, $C_{22}H_{24}N_2O_2$ (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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Experimental. Compound prepared by reacting $C_{22}H_{24}N_2O_2$ with $FeCl_3$ in acetone and recrystallized as dark-red prisms from the same solvent. D_m not measured. Crystal 0·50 × 0·40 × 0·35 mm. CAD-4 diffractometer, Mo $K\alpha$ radiation (graphite monochromator). Cell parameters from the setting angles for 25 reflections ($16 < \theta < 18°$). Data collected in ω–2θ scan mode, ω-scan width = (0·85 + 0·35tanθ)°, ω-scan speed 0·87–6·67° min^{-1}, $1·5 < \theta < 25°$, $\pm h$, k, l. Two standards monitored every 1 h of exposure time showed only minor fluctuations in intensities (merging R = 0·0083). 4016 reflections measured, 3532 unique, 2678 observed [$F_o > 3\sigma(F_o)$]. Empirical absorption corrections (transmission factors 0·670–0·999) applied. Fe atom located from Patterson map and other atoms from difference electron density maps. Full-matrix least-squares refinement on F finally converged at $R = 0·043$, $R_w = 0·053$, $S = 1·992$ for 2676 observed ($R = 0·066$, $R_w = 0·055$, $S = 1·333$ for all 3532) data; $w = 1·7472/[\sigma^2(F_o) + 0·0008F_o^2]$, $\sigma(F_o)$ from counting statistics. All H atoms located from difference maps were isotropic, non-H atoms anisotropic. 341 parameters refined. $(\Delta/\sigma)_{max} = 0·02$ [z for H(9B)]. Max. values of residual peaks and troughs in final difference map +0·6 and −0·7 e Å^{-3}. Programs used: *SHELX*80 (Sheldrick, 1980), *XANADU* (Roberts & Sheldrick, 1975) and *SNOOPI* (Davies, 1983) on a VAX 11/750 computer. Neutral-atom scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) for the non-H atoms respectively; f' and f'' values for Fe from Cromer & Liberman (1970).

Discussion. Fractional coordinates of the non-H atoms, and selected interatomic distances and angles are given in Tables 1 and 2.* The molecular structure and the atom numbering are shown in Fig. 1. Molecular packing is shown in Fig. 2. The metal atom is bonded to the Cl$^-$ and to the four N_2O_2 donor atoms of the bzen ligand in a distorted square-pyramidal environment. The basic metal coordination, and the Fe—Cl and Fe—O/N bond lengths [Fe—Cl 2·236 (3); Fe—O 1·906 (4), 1·925 (4); Fe—N 2·051 (5), 2·056 (5) Å] are close to those in the related pentacoordinate salen [*N*,*N*′-ethylenebis(salicylideneaminato)] complexes [FeCl(salen)] [Fe—Cl 2·238 (4); Fe—O 1·88 (1), 1·89 (1); Fe—N 2·06 (1), 2·10 (1) Å] (Gerloch & Mabbs, 1967) and [Fe_2(salen)$_2$O] [Fe—O(salen) 1·89 (2)–1·95 (2); Fe—N 2·09 (2)–2·11 (2) Å] (Gerloch, McKenzie & Towl, 1969). The N_2O_2 donor atoms at the base of the pyramid show tetrahedral distortions with atomic deviations of ~0·23 Å. The Fe atom is displaced from the mean plane of these four atoms by

Table 1. *Fractional coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2 ×10^3) *for the non-hydrogen atoms with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}
Fe	4480·5 (3)	1712·3 (3)	1359·9 (4)	30·4
Cl	4913·9 (8)	2681·3 (6)	2885·8 (10)	53·8
O(1)	3099 (2)	1256 (1)	1594 (3)	40
O(2)	5161 (2)	712 (2)	2075 (2)	41
N(1)	3765 (2)	2557 (2)	72 (3)	37
N(2)	5525 (2)	1662 (2)	−113 (3)	33
C(1)	2156 (2)	1572 (2)	1304 (3)	33
C(2)	1979 (3)	2303 (2)	616 (4)	39
C(3)	2772 (2)	2780 (2)	−10 (3)	36
C(4)	4600 (3)	2989 (3)	−599 (5)	47
C(5)	5340 (3)	2328 (2)	−1113 (4)	41
C(6)	6225 (2)	1071 (2)	−318 (3)	34
C(7)	6430 (3)	390 (2)	560 (3)	37
C(8)	5922 (2)	240 (2)	1686 (3)	30
C(9)	6866 (4)	1080 (3)	−1505 (5)	54
C(10)	2382 (4)	3560 (3)	−743 (6)	58
C(11)	6216 (2)	−479 (2)	2576 (3)	32
C(12)	7031 (3)	−1053 (2)	2341 (4)	42
C(13)	7273 (3)	−1696 (3)	3211 (5)	55
C(14)	6725 (4)	−1788 (3)	4310 (5)	59
C(15)	5906 (4)	−1231 (3)	4567 (4)	58
C(16)	5661 (3)	−587 (2)	3688 (4)	44
C(17)	1267 (2)	1058 (2)	1779 (3)	36
C(18)	260 (3)	1042 (3)	1124 (4)	52
C(19)	−520 (3)	510 (3)	1569 (5)	66
C(20)	−332 (3)	7 (3)	2638 (5)	65
C(21)	645 (3)	32 (3)	3300 (6)	62
C(22)	1442 (3)	549 (2)	2862 (4)	47

Table 2. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Cl—Fe	2·236 (3)	O(1)—Fe	1·906 (4)
O(2)—Fe	1·925 (4)	N(1)—Fe	2·051 (5)
N(2)—Fe	2·056 (5)	C(5)—C(4)	1·513 (7)
C(1)—O(1)	1·305 (5)	C(8)—O(2)	1·294 (5)
C(3)—N(1)	1·294 (5)	C(6)—N(2)	1·309 (5)
C(4)—N(1)	1·456 (5)	C(5)—N(2)	1·480 (5)
C(2)—C(1)	1·367 (6)	C(8)—C(7)	1·370 (6)
C(3)—C(2)	1·430 (6)	C(7)—C(6)	1·418 (6)
C(11)—C(8)	1·491 (5)	C(17)—C(1)	1·485 (5)
C(10)—C(3)	1·513 (7)	C(9)—C(6)	1·496 (6)

O(1)—Fe—Cl	111·1 (2)	O(2)—Fe—Cl	102·3 (2)
N(1)—Fe—Cl	94·8 (2)	N(2)—Fe—Cl	113·6 (2)
O(2)—Fe—O(1)	91·6 (2)	N(2)—Fe—N(1)	80·0 (2)
N(1)—Fe—O(1)	87·7 (2)	N(2)—Fe—O(2)	87·7 (2)
N(1)—Fe—O(2)	161·9 (1)	N(2)—Fe—O(1)	134·4 (1)
C(1)—O(1)—Fe	130·2 (2)	C(8)—O(2)—Fe	132·7 (2)
C(3)—N(1)—Fe	127·2 (3)	C(6)—N(2)—Fe	127·4 (3)
C(4)—N(1)—Fe	108·0 (3)	C(5)—N(2)—Fe	113·6 (3)
C(4)—N(1)—C(3)	124·0 (4)	C(6)—N(2)—C(5)	118·6 (4)
C(2)—C(1)—O(1)	124·2 (4)	C(7)—C(8)—O(2)	123·1 (4)
C(17)—C(1)—O(1)	113·8 (4)	C(11)—C(8)—O(2)	114·4 (4)
C(17)—C(1)—C(2)	122·0 (4)	C(11)—C(8)—C(7)	122·5 (4)
C(3)—C(2)—C(1)	125·5 (4)	C(8)—C(7)—C(6)	126·2 (4)
C(2)—C(3)—N(1)	121·3 (4)	C(7)—C(6)—N(2)	122·8 (4)
C(10)—C(3)—N(1)	122·6 (4)	C(9)—C(6)—N(2)	121·3 (4)
C(10)—C(3)—C(2)	116·1 (4)	C(9)—C(6)—C(7)	115·9 (4)
C(5)—C(4)—N(1)	108·2 (4)	C(4)—C(5)—N(2)	109·1 (4)

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* Anisotropic temperature factors for the non-H atoms, coordinates, bond lengths and angles involving the H atoms, dimensions of the phenyl rings, selected least-squares-planes' and dihedral-angle calculations, and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39814 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0·525 (4) Å towards the apical Cl^-. The two chemically equivalent halves of the bzen ligand are bent away from the axial Cl^- and twisted unsymmetrically, due mainly to several intramolecular short contacts [Cl···O(1) 3·420 (6), Cl···O(2) 3·246 (6), Cl···N(1) 3·158 (6), Cl···N(2) 3·592 (6), Cl···C(4) 3·605 (6) Å] and the conformational requirement of the ethylenediamine bridge with a fairly large NCCN torsion angle, 41·3 (4)°. The Fe atom is placed 0·386 (4) Å above the plane of the atoms O(1),N(1),C(1)–C(3) (chelate 1) and 0·070 (4) Å below the plane of the atoms O(2),-N(2),C(6)–C(8) (chelate 2). The two chelate planes mutually inclined at 37·6 (4)° are unsymmetrically inclined to the coordination plane at 12·8 (4)° (chelate 1) and 26·2 (4)° (chelate 2). These values together with unequal displacements of the two ethylenediamine C atoms [C(4) 0·477 (5), C(5) –0·329 (5) Å] from the coordination plane suggest the conformation as an asymmetric unbrella (Calligaris, Nardin & Randaccio, 1972).

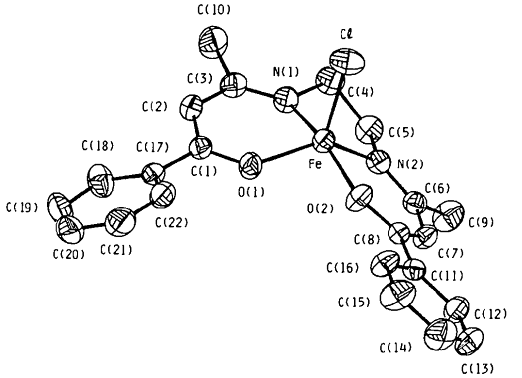

Fig. 1. A general view of the molecular structure of [$FeCl(C_{22}H_{22}N_2O_2)$] showing the atom numbering.

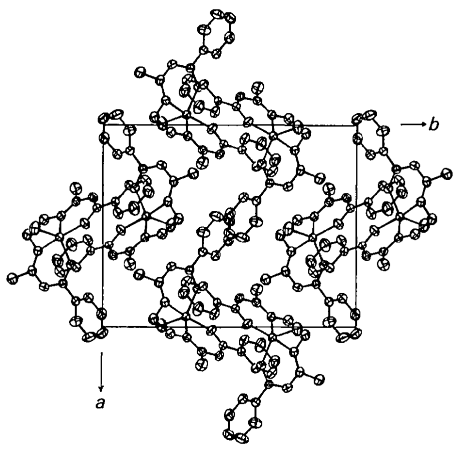

Fig. 2. Molecular packing viewed along **c**.

The phenyl groups are each planar and have normal bond lengths and angles. However, one group is significantly noncoplanar whilst the other is nearly coplanar with the respective chelate rings, as indicated by the torsion angles O(1)–C(2)–C(17)–C(22) 26·8 (4) and O(2)–C(8)–C(11)–C(16) 1·1 (4)°. This situation is in sharp contrast with that in the square-planar bzen complexes of Co^{II} (Haider, Hashem, Malik & Hursthouse, 1980), Ni^{II} (Malatesta & Mugnoli, 1981), Cu^{II} and Pd^{II} (Malik, Haider, Hashem & Hursthouse, 1985), where the corresponding torsion angles are all ~11–16°. Discrete monomeric molecules are held together in the crystal mostly by van der Waals forces. The minimum distance between two non-H atoms in the neighbouring molecules is 3·487 (7) Å [C(7)···O(1) (at $1-x$, $-y$, $-z$)]. In addition, the Cl atom is involved in a weak, intermolecular H bond [C(15)–H(15)···Cl (at $1-x$, $-y$, $1-z$)] with dimensions: C(15)–H(15) 1·02 (5), C(15)···Cl 3·667 (5), H(15)···Cl 2·84 (5) Å, ∠C(15)–H(15)···Cl 138 (5)°.

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