Cl(1)	0.0210 (2)	0.2650 (2)	0.6341 (2)	0.167 (1)
Cl(2)	0.0491 (2)	0.0716 (3)	0.6059 (2)	0.226 (2)
C(51)	0.0790 (5)	0.1722 (7)	0.6512 (5)	0.132 (4)

Table 2. *Geometric parameters* (Å, °)

Ir(1)—Ir(2)	3.307 (1)	N(3)—N(4)	1.376 (6)
Ir(1)—N(1)	2.075 (4)	N(3)—C(8)	1.337 (7)
Ir(1)—N(3)	2.091 (4)	N(4)—C(9)	1.332 (7)
Ir(2)—N(2)	2.068 (4)	O(1)—C(1)	1.174 (7)
Ir(2)—N(4)	2.073 (4)	O(2)—C(2)	1.165 (7)
Ir(1)—C(1)	1.797 (6)	C(3)—C(5)	1.371 (8)
Ir(2)—C(2)	1.810 (6)	C(3)—C(6)	1.493 (9)
Ir(1)—P(1)	2.224 (1)	C(4)—C(5)	1.377 (8)
Ir(2)—P(2)	2.224 (1)	C(4)—C(7)	1.494 (8)
P(1)—O(3)	1.629 (4)	C(8)—C(10)	1.365 (9)
P(2)—O(4)	1.633 (4)	C(8)—C(11)	1.488 (9)
N(1)—N(2)	1.370 (6)	C(9)—C(10)	1.377 (9)
N(1)—C(3)	1.349 (7)	C(9)—C(12)	1.493 (9)
N(2)—C(4)	1.334 (7)		
P(1)—Ir(1)—N(3)	92.3 (1)	Ir(2)—P(2)—C(39)	117.8 (2)
P(1)—Ir(1)—C(1)	91.0 (2)	Ir(1)—N(1)—N(2)	117.9 (3)
N(1)—Ir(1)—N(3)	85.1 (2)	Ir(1)—N(1)—C(3)	135.1 (3)
N(1)—Ir(1)—C(1)	91.6 (2)	C(3)—N(1)—N(2)	106.8 (4)
P(1)—Ir(1)—N(1)	177.4 (1)	Ir(2)—N(2)—N(1)	117.1 (3)
N(3)—Ir(1)—C(1)	176.3 (2)	Ir(2)—N(2)—C(4)	133.0 (3)
P(2)—Ir(2)—N(2)	92.5 (1)	C(4)—N(2)—N(1)	109.3 (4)
P(2)—Ir(2)—C(2)	91.8 (2)	Ir(1)—N(3)—N(4)	117.2 (3)
N(2)—Ir(2)—N(4)	83.1 (2)	Ir(1)—N(3)—C(8)	134.3 (4)
P(2)—Ir(2)—N(4)	174.2 (1)	C(8)—N(3)—N(4)	108.4 (4)
N(2)—Ir(2)—C(2)	174.1 (2)	C(9)—N(4)—N(3)	107.4 (4)
N(4)—Ir(2)—C(2)	92.9 (2)	Ir(2)—N(4)—N(3)	118.0 (3)
Ir(1)—C(1)—O(1)	179.3 (5)	Ir(2)—N(4)—C(9)	134.5 (4)
Ir(2)—C(2)—O(2)	178.0 (5)	C(5)—C(3)—N(1)	109.2 (5)
Ir(1)—P(1)—O(3)	119.3 (1)	C(5)—C(4)—N(2)	108.2 (5)
Ir(1)—P(1)—C(27)	116.5 (2)	C(4)—C(5)—C(3)	106.5 (5)
Ir(1)—P(1)—C(45)	116.4 (2)	C(10)—C(8)—N(3)	108.6 (5)
Ir(2)—P(2)—O(4)	119.7 (1)	C(10)—C(9)—N(4)	109.1 (5)
Ir(2)—P(2)—C(33)	116.2 (2)	C(9)—C(10)—C(8)	106.5 (5)

Weights were taken as $1/\sigma^2(F_o^2)$; variances $[\sigma^2(F_o^2)]$ derived from counting statistics plus an additional term, $(0.014I)^2$; variances of the merged data by propagation of error plus another additional term, $(0.014\langle I\rangle)^2$. Goodness of fit for merging data was 1.02; R_{merge} for duplicates, 0.020. Dispersion corrections were taken from Cromer & Waber (1974). The final R for $F_o^2 > 3\sigma$ was 0.0235; the final wR, 0.0022. Since the calculated absorption correction increased the goodness of fit for merging, an absorption coefficient corresponding to 30% of the calculated value was used.

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 55976 (41 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1021]

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Structure of [*N*,*N*′-*o*-Phenylenebis(salicylideneaminato)]iron(III) Chloride as a Five-Coordinate Monomer

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Abstract

The crystal contains three independent five-coordinate monomers of chloro{2,2′-[*o*-phenylenebis(nitrilomethylidyne)]diphenolato-*N*,*N*′,*O*,*O*′}iron(III). The distances Fe(1)—Fe(1*A*), Fe(1)—Fe(1*B*) and Fe(1*A*)—Fe(1*B*) are 7.175 (1), 7.683 (1) and 7.207 (1) Å, respectively. The planes of the ligand groups of the two neighbouring molecules bend away from each other.

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Comment

Suitable crystals were obtained directly from the synthesis of the title compound. Two solutions, *N,N'-o*-phenylenebis(salicylideneamine) in THF and $FeCl_2.4H_2O$ in methanol, were prepared and heated to boiling temperature. The mixture of the two solutions was then refluxed for 4 h. Crystals were obtained after two to three days.

Complexes of transition-metal ions with Schiff bases provide an increasingly large class of compounds of both stereochemical and magnetochemical interest. We have reported previously the structures of several dimeric Schiff-base complexes of iron(III) (Elmali, Atakol, Svoboda & Fuess, 1992, 1993; Elmali, Elerman, Svoboda & Fuess, 1993). The present structure is a five-coordinate monomer of iron(III). The structures of Fe(salen)Cl [salen = *N,N'*-ethylenebis(salicylideneaminato)] were, however, reported to be a five-coordinated monomer and a six-coordinated dimer (Gerloch & Mabbs, 1967).

The coordination of iron(III) in the three molecules is essentially square pyramidal with the metal atom 0.52 (1), 0.52 (1) and 0.53 (1) Å, respectively, above the best plane defined by the Schiff-base donor atoms. The Fe—Cl, Fe—O and Fe—N bond lengths are almost identical in all of the molecules and are consistent with the corresponding values in the monomeric Fe(salen)Cl. The angles O(1)—Fe—Cl(1), N(16)—Fe—O(1), O(24)—Fe—N(9) and O(24)—Fe—Cl(1) do, however, show significant differences.

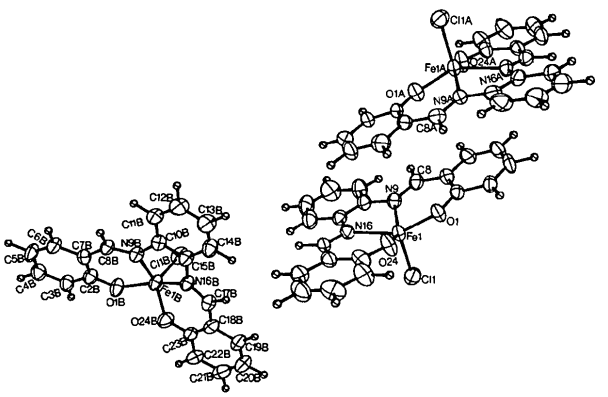

Fig. 1. The molecular structure of the title compound; anisotropic ellipsoids represent 50% probability boundaries. H atoms are drawn as spheres of arbitrary radii.

Experimental

Crystal data

$[Fe(C_{20}H_{14}N_2O_2)Cl]$	D_x = 1.36 Mg m^{-3}
M_r = 405.64	Mo $K\alpha$ radiation
Triclinic	λ = 0.71069 Å
$P\bar{1}$	Cell parameters from 52 reflections
a = 15.106 (7) Å	θ = 17.55–20.54°
b = 13.570 (5) Å	μ = 0.915 mm^{-1}
c = 7.386 (3) Å	T = 303 K
α = 101.55 (2)°	Prism
β = 94.18 (2)°	0.18 × 0.4 × 0.6 mm
γ = 92.49 (2)°	Black
V = 1476.83 Å^3	
Z = 3	

Data collection

Stoe Stadi-4 diffractometer	7844 observed reflections $[F>2.0\sigma(F)]$
$\omega/2\theta$ scans	θ_{max} = 23°
Absorption correction: Gaussian by integration from crystal shape	$h = -16 \rightarrow 16$
	$k = -14 \rightarrow 14$
T_{min} = 0.9118, T_{max} = 0.9998	$l = -8 \rightarrow 8$
8245 measured reflections	3 standard reflections
8077 independent reflections	frequency: 120 min
	intensity variation: 4%

Refinement

Refinement on F	$\Delta\rho_{max}$ = 0.99 e Å^{-3}
Final R = 0.0484	$\Delta\rho_{min}$ = −0.62 e Å^{-3}
wR = 0.0466	Extinction correction: empirical isotropic
S = 1.030	Extinction coefficient: 0.00263
7844 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)
705 parameters	
H-atom parameters not refined	
$w = 1/[\sigma^2(F)+0.0001F^2]$	
$(\Delta/\sigma)_{max}$ = 0.2	

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1986). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Fe(1)	0.3440	0.8203	0.5359	0.039 (1)
Cl(1)	0.3122 (1)	0.7529 (1)	0.7768 (3)	0.062 (1)
O(1)	0.2928 (3)	0.9446 (3)	0.5592 (6)	0.048 (2)
C(2)	0.3198 (4)	1.0377 (4)	0.6427 (8)	0.041 (3)
C(3)	0.2574 (4)	1.1156 (5)	0.6591 (9)	0.046 (3)
C(4)	0.2825 (4)	1.2107 (4)	0.742 (1)	0.054 (3)
C(5)	0.3713 (4)	1.2375 (5)	0.819 (1)	0.054 (3)
C(6)	0.4325 (4)	1.1671 (4)	0.8030 (9)	0.049 (3)
C(7)	0.4102 (4)	1.0668 (4)	0.7146 (8)	0.040 (3)
C(8)	0.4774 (4)	0.9965 (4)	0.6981 (8)	0.042 (3)
N(9)	0.4668 (3)	0.8996 (4)	0.6271 (7)	0.041 (2)
C(10)	0.5411 (4)	0.8383 (4)	0.6094 (9)	0.043 (3)
C(11)	0.6258 (4)	0.8697 (5)	0.698 (1)	0.061 (3)
C(12)	0.6949 (5)	0.8067 (6)	0.665 (1)	0.074 (4)
C(13)	0.6793 (5)	0.7127 (6)	0.552 (1)	0.071 (4)
C(14)	0.5946 (5)	0.6800 (5)	0.463 (1)	0.061 (4)
C(15)	0.5255 (4)	0.7445 (5)	0.4954 (9)	0.046 (3)
N(16)	0.4370 (3)	0.7211 (3)	0.4155 (7)	0.041 (2)
C(17)	0.4158 (4)	0.6385 (4)	0.2862 (9)	0.046 (3)
C(18)	0.3310 (4)	0.6131 (4)	0.1935 (9)	0.045 (3)
C(19)	0.3213 (5)	0.5215 (5)	0.067 (1)	0.060 (4)
C(20)	0.2384 (6)	0.4887 (5)	−0.0362 (1)	0.068 (4)
C(21)	0.1685 (5)	0.5503 (6)	−0.013 (1)	0.069 (4)
C(22)	0.1765 (5)	0.6415 (5)	0.114 (1)	0.060 (4)
C(23)	0.2568 (4)	0.6747 (4)	0.2168 (9)	0.050 (3)

O(24)	0.2628 (3)	0.7619 (3)	0.3338 (7)	0.056 (2)
Fe(1A)	0.5081 (1)	1.3256 (1)	0.4330 (1)	0.038 (1)
Cl(1A)	0.5424 (1)	1.3890 (1)	0.1887 (3)	0.058 (1)
O(1A)	0.5592 (3)	1.1998 (3)	0.4243 (6)	0.049 (2)
C(2A)	0.5341 (4)	1.1072 (4)	0.3409 (8)	0.039 (3)
C(3A)	0.5954 (4)	1.0347 (4)	0.3349 (9)	0.046 (3)
C(4A)	0.5722 (5)	0.9361 (5)	0.240 (1)	0.056 (3)
C(5A)	0.4899 (5)	0.9092 (5)	0.154 (1)	0.058 (3)
C(6A)	0.4264 (5)	0.9801 (5)	0.1657 (9)	0.056 (3)
C(7A)	0.4472 (4)	1.0815 (4)	0.2571 (8)	0.041 (3)
C(8A)	0.3769 (4)	1.1500 (5)	0.2677 (9)	0.046 (3)
N(9A)	0.3868 (3)	1.2453 (3)	0.3372 (7)	0.038 (2)
C(10A)	0.3126 (4)	1.3058 (4)	0.3442 (9)	0.046 (3)
C(11A)	0.2304 (4)	1.2791 (5)	0.246 (1)	0.064 (4)
C(12A)	0.1642 (4)	1.3457 (6)	0.271 (1)	0.071 (4)
C(13A)	0.1761 (4)	1.4374 (6)	0.390 (1)	0.068 (4)
C(14A)	0.2578 (4)	1.4668 (5)	0.483 (1)	0.054 (3)
C(15A)	0.3277 (3)	1.4019 (4)	0.4634 (9)	0.041 (3)
N(16A)	0.4144 (3)	1.4238 (4)	0.5470 (7)	0.041 (2)
C(17A)	0.4337 (4)	1.5040 (5)	0.6702 (9)	0.045 (3)
C(18A)	0.5208 (4)	1.5367 (4)	0.7668 (9)	0.044 (3)
C(19A)	0.5319 (5)	1.6329 (5)	0.889 (1)	0.056 (3)
C(20A)	0.6096 (5)	1.6664 (5)	0.981 (1)	0.064 (4)
C(21A)	0.6828 (5)	1.6085 (5)	0.956 (1)	0.058 (3)
C(22A)	0.6753 (4)	1.5156 (5)	0.843 (1)	0.055 (3)
C(23A)	0.5952 (4)	1.4781 (4)	0.7417 (9)	0.044 (3)
O(24A)	0.5908 (3)	1.3880 (3)	0.6347 (6)	0.051 (2)
Fe(1B)	0.9557 (1)	0.1661 (1)	0.5114 (1)	0.044 (1)
Cl(1B)	1.0439 (1)	0.2854 (2)	0.6992 (3)	0.062 (1)
O(1B)	1.0285 (3)	0.0611 (4)	0.4199 (6)	0.061 (2)
C(2B)	1.0659 (4)	0.0325 (5)	0.2651 (9)	0.047 (3)
C(3B)	1.1235 (4)	−0.0458 (5)	0.249 (1)	0.054 (3)
C(4B)	1.1636 (4)	−0.0769 (5)	0.088 (1)	0.054 (3)
C(5B)	1.1503 (5)	−0.0313 (6)	−0.0610 (1)	0.062 (4)
C(6B)	1.0931 (4)	0.0433 (5)	−0.052 (1)	0.053 (3)
C(7B)	1.0503 (4)	0.0781 (5)	0.1090 (9)	0.043 (3)
C(8B)	0.9942 (4)	0.1600 (5)	0.1104 (9)	0.044 (3)
N(9B)	0.9541 (3)	0.2051 (4)	0.2516 (7)	0.042 (2)
C(10B)	0.9020 (4)	0.2892 (5)	0.2391 (9)	0.041 (2)
C(11B)	0.9142 (4)	0.3517 (5)	0.1120 (9)	0.052 (3)
C(12B)	0.8591 (4)	0.4300 (6)	0.115 (1)	0.065 (4)
C(13B)	0.7937 (5)	0.4469 (6)	0.238 (1)	0.072 (4)
C(14B)	0.7857 (4)	0.3904 (5)	0.365 (1)	0.059 (3)
C(15B)	0.8398 (4)	0.3094 (5)	0.3687 (9)	0.045 (3)
N(16B)	0.8378 (3)	0.2437 (4)	0.4954 (7)	0.042 (2)
C(17B)	0.7732 (4)	0.2422 (5)	0.6037 (9)	0.044 (3)
C(18B)	0.7688 (4)	0.1823 (5)	0.7383 (9)	0.046 (3)
C(19B)	0.7001 (5)	0.2013 (6)	0.860 (1)	0.057 (4)
C(20B)	0.6934 (5)	0.1498 (6)	1.002 (1)	0.069 (4)
C(21B)	0.7502 (5)	0.0742 (6)	1.018 (1)	0.061 (4)
C(22B)	0.8169 (4)	0.0513 (6)	0.895 (1)	0.056 (4)
C(23B)	0.8285 (4)	0.1080 (5)	0.7597 (9)	0.045 (3)
O(24B)	0.8946 (3)	0.0877 (3)	0.6524 (7)	0.060 (2)

Table 2. *Geometric parameters* (Å, °)

Fe(1)—Cl(1)	2.230 (2)	Fe(1A)—N(16A)	2.091 (5)
Fe(1)—O(1)	1.868 (4)	Fe(1A)—O(24A)	1.904 (4)
Fe(1)—N(9)	2.099 (4)	Fe(1B)—Cl(1B)	2.228 (2)
Fe(1)—N(16)	2.108 (5)	Fe(1B)—O(1B)	1.885 (5)
Fe(1)—O(24)	1.881 (4)	Fe(1B)—N(9B)	2.088 (5)
Fe(1A)—Cl(1A)	2.232 (2)	Fe(1B)—N(16B)	2.116 (5)
Fe(1A)—O(1A)	1.895 (4)	Fe(1B)—O(24B)	1.889 (5)
Fe(1A)—N(9A)	2.090 (4)		
O(1)—Fe(1)—Cl(1)	108.5 (2)	N(16A)—Fe(1A)—N(9A)	76.8 (2)
N(9)—Fe(1)—Cl(1)	103.1 (2)	O(24A)—Fe(1A)—Cl(1A)	106.7 (2)
N(9)—Fe(1)—O(1)	87.8 (2)	O(24A)—Fe(1A)—O(1A)	91.2 (2)
N(16)—Fe(1)—Cl(1)	101.0 (2)	O(24A)—Fe(1A)—N(9A)	149.4 (2)
N(16)—Fe(1)—O(1)	149.1 (2)	O(24A)—Fe(1A)—N(16A)	88.0 (2)
N(16)—Fe(1)—N(9)	76.6 (2)	O(1B)—Fe(1B)—Cl(1B)	106.7 (1)
O(24)—Fe(1)—Cl(1)	107.9 (2)	N(9B)—Fe(1B)—Cl(1B)	105.0 (1)
O(24)—Fe(1)—O(1)	92.0 (2)	N(9B)—Fe(1B)—O(1B)	87.6 (2)
O(24)—Fe(1)—N(9)	147.4 (2)	N(16B)—Fe(1B)—Cl(1B)	100.2 (2)
O(24)—Fe(1)—N(16)	87.5 (2)	N(16B)—Fe(1B)—O(1B)	151.6 (2)
O(1A)—Fe(1A)—Cl(1A)	110.0 (2)	N(16B)—Fe(1B)—N(9B)	76.5 (2)
N(9A)—Fe(1A)—Cl(1A)	102.4 (2)	O(24B)—Fe(1B)—Cl(1B)	109.9 (2)
N(9A)—Fe(1A)—O(1A)	87.6 (2)	O(24B)—Fe(1B)—O(1B)	92.9 (2)
N(16A)—Fe(1A)—Cl(1A)	101.9 (2)	O(24B)—Fe(1B)—N(9B)	143.4 (2)
N(16A)—Fe(1A)—O(1A)	146.8 (2)	O(24B)—Fe(1B)—N(16B)	86.5 (2)

The *x*, *y* and *z* coordinates of Fe(1) were fixed to define the origin of the structure. All H atoms were located geometrically (C—H 0.98 Å). Refinement was by the full-matrix least-squares method. The polarity was checked by inversion of all parameters; the refinement converged to identical *R* values in both cases. The polarity presented here was chosen arbitrarily.

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71052 (114 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1030]

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Redetermination of the Structure of μ_6-Acetonato-1:2:3κ^3C^1;4:5:6κ^3C^3-bis[nonacarbonyl-1κ^3C,2κ^3C,3κ^3C-*triangulo*-tricobalt(3 *Co–Co*)] at 128 K

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Abstract

The structure of the carbonyl-bridged dicluster compound $OC[CCo_3(CO)_9]_2$ has been redetermined from diffractometer data recorded at 128 K. The broad