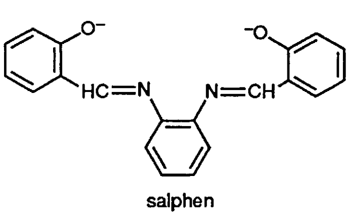
Cubane Structure of Sodium Derivatives of Tetradentate Schiff Bases†

Euro Solari,[a] **Stefania De Angelis,**[a] **Carlo Floriani,***[,a] **Angiola Chiesi-Villa**[b] **and Corrado Rizzoli**[b]

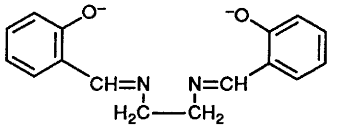
[a] *Section de Chimie, Université de Lausanne, Place du Château 3, CH-1005 Lausanne, Switzerland*
[b] *Istituto di Strutturistica Chimica, Centro di Studio per la Strutturistica Diffrattometrica del CNR, Università di Parma, I-43100 Parma, Italy*

Reaction of sodium hydride in thf (tetrahydrofuran) with H_2salphen [*N,N'*-*o*-phenylenebis(salicylideneimine)] or H_2salen [*N,N'*-ethylenebis(salicylideneimine)] gave the corresponding sodium derivatives Na_2(salphen)(thf)$_n$ and Na_2(salen)(thf)$_n$. Recrystallization from 1,2-dimethoxyethane (dme) gave the stable solvated forms [Na_4(salphen)$_2$(dme)$_2$] **1** and [Na_4(salen)$_2$(dme)$_2$] **2**. From the X-ray analysis both have very similar structures with a cubane skeleton of alternate sodium and oxygen atoms. Crystallographic details: complex **1**, space group $P2_1/c$, $a = 12.045(1)$, $b = 25.851(2)$, $c = 16.467(1)$ Å, $\alpha = \gamma = 90°$, $\beta = 107.61(1)°$, $Z = 4$ and $R = 0.079$ for 2118 independent observed reflections; **2**, space group $P2_1/n$, $a = 20.493(2)$, $b = 21.682(2)$, $c = 20.538(2)$ Å, $\alpha = 90°$, $\beta = 110.53(1)°$, $Z = 8$ and $R = 0.071$ for 5054 independent observed reflections.

Tetradentate Schiff-base ligands derived from the condensation of salicylaldehyde and *o*-phenylenediamine or ethylenediamine, H_2salphen [*N,N'*-*o*-phenylenebis(salicylideneimine)] and H_2salen [*N,N'*-ethylenebis(salicylideneimine)], have been almost ubiquitous polydentate ligands in co-ordination chemistry for over a century.[1] Synthetic methods have moved over the years from aqueous solutions[2] to organic solvents.[3] In the latter case a widely used methodology employed the alkali-metal derivatives of Schiff bases, formed from the corresponding alkali-metal hydrides.[3] Reactions are usually carried out *in situ*, while a more direct method should lead to the isolation of a crystalline form of the salt with a well defined stoichiometry. At the same time it may be very informative to know the structure of such a starting material and to take advantage of it. The bonding mode of the salphen and salen dianions to transition metals is very well known;[1] in contrast we know nothing of their bonding mode to alkali-metal cations.[4] Therefore, herein we describe a high yield synthesis of crystalline Na_2(salphen) and Na_2(salen), their solid-state structure being determined by X-ray crystallography.



salphen



salen

Results and Discussion

Reaction of H_2salphen or H_2salen with sodium hydride in thf (tetrahydrofuran) is probably the best method of generating the corresponding sodium salt, which can be used in transition-metal complexation. Recrystallization from 1,2-dimethoxyethane (dme) gave stable solvated forms having the stoichiometry reported in reactions (1) and (2). The use of other

$$H_2\text{salphen} + 2\text{NaH} \xrightarrow[-H_2]{\text{thf}} \xrightarrow{\text{dme}} [\text{Na}_4(\text{salphen})_2(\text{dme})_2] \quad (1)$$

$$H_2\text{salen} + 2\text{NaH} \xrightarrow[-H_2]{\text{thf}} \xrightarrow{\text{dme}} [\text{Na}_4(\text{salen})_2(\text{dme})_2] \quad (2)$$

metallating agents should be avoided, *e.g.*, alkali metals lead to partial hydrogenation of imino groups. Complexes **1** and **2** have been isolated as crystals suitable for an X-ray analysis. They can be investigated also in solution by spectroscopy such as NMR but we cannot be certain that the molecular complexity remains intact in some co-ordinating solvents, where the solubility is reasonable, for **1** and **2**. The structures of compounds **1** and **2** are shown in Figs. 1 and 2, and selected bond distances and angles listed in Table 1.

The structure of compounds **1** and **2** consists of a discrete cubane skeleton of alternate sodium and oxygen atoms derived from the Schiff-base dianion. In the asymmetric unit of complex **2** there are two crystallographically independent molecules (A and B) having very close geometries except for the conformation of the N $\cdots$ N five-membered chelation rings. Hereinafter we will refer to molecule A only. Bond distances and angles within the cubes are almost regular

---

† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1991, Issue 1, pp. xviii–xxii.

Table 1 Selected bond distances (Å) and angles (°)

Complex **1**

Na(1)–O(1)	2.252(10)	Na(2)–O(1)	2.369(10)	Na(3)–O(2)	2.252(9)	Na(4)–O(1)	2.362(9)
Na(1)–O(2)	2.259(10)	Na(2)–O(2)	2.354(10)	Na(3)–O(3)	2.291(9)	Na(4)–O(3)	2.385(10)
Na(1)–O(4)	2.332(9)	Na(2)–O(3)	2.354(8)	Na(3)–O(4)	2.265(10)	Na(4)–O(4)	2.366(10)
Na(1)–N(1)	2.378(11)	Na(2)–O(5)	2.356(12)	Na(3)–N(3)	2.309(10)	Na(4)–O(7)	2.371(13)
Na(1)–N(2)	2.370(11)	Na(2)–O(6)	2.401(12)	Na(3)–N(4)	2.349(12)	Na(4)–O(8)	2.349(10)

N(1)–Na(1)–N(2)	69.6(4)	O(5)–Na(2)–O(6)	69.8(4)	N(3)–Na(3)–N(4)	71.4(4)	O(7)–Na(4)–O(8)	72.2(4)
O(2)–Na(1)–O(4)	93.4(3)	O(2)–Na(2)–O(3)	91.3(3)	O(3)–Na(3)–O(4)	97.1(3)	O(3)–Na(4)–O(4)	91.9(3)
O(1)–Na(1)–O(4)	93.6(3)	O(1)–Na(2)–O(3)	89.7(3)	O(2)–Na(3)–O(4)	95.4(4)	O(1)–Na(4)–O(4)	90.0(3)
O(1)–Na(1)–O(2)	93.7(3)	O(1)–Na(2)–O(2)	88.3(3)	O(2)–Na(3)–O(3)	95.6(3)	O(1)–Na(4)–O(3)	89.1(3)
Na(2)–O(1)–Na(4)	90.7(3)	Na(2)–O(2)–Na(3)	87.0(3)	Na(3)–O(3)–Na(4)	84.9(3)	Na(3)–O(4)–Na(4)	86.0(3)
Na(1)–O(1)–Na(4)	89.1(3)	Na(1)–O(2)–Na(3)	86.3(3)	Na(2)–O(3)–Na(4)	90.5(3)	Na(1)–O(4)–Na(4)	87.2(3)
Na(1)–O(1)–Na(2)	88.9(3)	Na(1)–O(2)–Na(2)	89.1(3)	Na(2)–O(3)–Na(3)	86.1(3)	Na(1)–O(4)–Na(3)	84.3(3)

Complex **2**

	Molecule A	Molecule B		Molecule A	Molecule B
Na(1)–O(1)	2.277(6)	2.268(6)	Na(3)–O(2)	2.329(8)	2.337(8)
Na(1)–O(2)	2.349(7)	2.314(7)	Na(3)–O(3)	2.398(6)	2.413(7)
Na(1)–O(4)	2.399(7)	2.356(7)	Na(3)–O(4)	2.348(6)	2.318(7)
Na(1)–N(1)	2.382(10)	2.366(10)	Na(3)–O(7)	2.454(9)	2.414(10)
Na(1)–N(2)	2.357(8)	2.349(8)	Na(3)–O(8)	2.306(17)	2.346(14)
Na(2)–O(1)	2.298(6)	2.329(6)	Na(4)–O(1)	2.296(7)	2.301(7)
Na(2)–O(2)	2.435(6)	2.416(7)	Na(4)–O(3)	2.280(7)	2.294(7)
Na(2)–O(3)	2.359(8)	2.374(8)	Na(4)–O(4)	2.273(6)	2.278(6)
Na(2)–O(5)	2.486(8)	2.453(8)	Na(4)–N(3)	2.400(11)	2.302(10)
Na(2)–O(6)	2.329(8)	2.366(8)	Na(4)–N(4)	2.312(8)	2.348(8)
N(1)–Na(1)–N(2)	70.2(3)	70.1(3)	O(7)–Na(3)–O(8)	66.8(4)	69.1(4)
O(2)–Na(1)–O(4)	91.4(2)	89.2(3)	O(3)–Na(3)–O(4)	90.4(2)	92.2(2)
O(1)–Na(1)–O(4)	89.0(2)	92.1(2)	O(2)–Na(3)–O(4)	93.2(2)	89.5(3)
O(1)–Na(1)–O(2)	94.4(2)	93.6(2)	O(2)–Na(3)–O(3)	90.2(2)	90.3(2)
O(5)–Na(2)–O(6)	68.9(3)	67.6(3)	N(3)–Na(4)–N(4)	71.2(4)	71.5(4)
O(2)–Na(2)–O(3)	88.6(2)	89.3(2)	O(3)–Na(4)–O(4)	95.4(2)	96.5(2)
O(1)–Na(2)–O(3)	90.8(2)	91.9(2)	O(1)–Na(4)–O(4)	91.7(2)	93.3(2)
O(1)–Na(2)–O(2)	91.6(2)	89.5(2)	O(1)–Na(4)–O(3)	92.9(2)	94.8(3)
Na(2)–O(1)–Na(4)	88.7(2)	87.0(2)	Na(3)–O(3)–Na(4)	86.4(2)	84.4(2)
Na(1)–O(1)–Na(4)	90.7(2)	88.0(2)	Na(2)–O(3)–Na(4)	87.6(2)	86.1(3)
Na(1)–O(1)–Na(2)	89.4(2)	90.0(2)	Na(2)–O(3)–Na(3)	90.5(2)	89.8(3)
Na(2)–O(2)–Na(3)	90.3(2)	90.6(2)	Na(3)–O(4)–Na(4)	87.8(2)	86.9(2)
Na(1)–O(2)–Na(3)	88.5(2)	90.9(2)	Na(1)–O(4)–Na(4)	88.2(2)	86.4(2)
Na(1)–O(2)–Na(2)	84.5(2)	86.8(2)	Na(1)–O(4)–Na(3)	86.9(2)	90.3(3)

Table 2 Comparison of structural parameters within the M(Schiff base) units

		Complex **2**	
	Complex **1**	Molecule A	Molecule B
Folding[a] along the N(1) · · · O(1) line/°	4.0(6)	17.4(2)	16.8(2)
Folding along the N(2) · · · O(2) line/°	3.8(3)	6.4(2)	7.4(2
Folding along the N(3) · · · O(3) line/°	3.2(3)	7.8(3)	0.6(3)
Folding along the N(4) · · · O(4) line/°	2.3(3)	4.8(2)	4.4(2)
Angle between M–N(1)–O(1) and M–N(2)–O(2) planes/°	117.8(6)	114.2(2)	115.6(3)
Angle between M–N(3)–O(3) and M–N(4)–O(4) planes/°	123.1(3)	118.3(3)	120.3(3)
N(1)N(2) torsion angle[b]/°	3.1(12)	−44.3(11)	−38.4(12)
N(3)N(4) torsion angle[c]/°	−3.4(13)	43.1(13)	−36.2(19)
Distance of C(8) from the M–N(1)–N(2) plane/Å	0.757(8)	0.374(11)	0.467(11)
Distance of C(13) (**1**) or C(9) (**2**) from the M–N(1)–N(2) plane/Å	0.778(8)	0.890(12)	0.891(11)
Distance of C(28) from the M–N(3)–N(4) plane/Å	0.756(8)	−0.324(12)	0.762(17)
Distance of C(33) (**1**) or C(29) (**2**) from the M–N(3) plane/Å	0.720(8)	−0.822(13)	0.381(16)
Distance of Na(1) from the N_2O_2 core/Å	0.961(4)	0.981(4)	0.962(4)
Distance of Na(3) from the N_2O_2 core/Å	0.846(5)		
Distance of Na(4) from the N_2O_2 core/Å		0.910(4)	0.894(4)

[a] The folding is defined as the dihedral angle between the NaNO and OC_3N planes of a six-membered chelation ring. [b] N(1)–C(8)–C(13)–N(2) for **1**, N(1)–C(8)–C(9)–N(2) for **2**. [c] N(3)–C(28)–C(33)–N(4) for **1**, N(3)–C(28)–C(29)–N(4) for **2**.

(Table 1). The Na–O and Na–N distances fall in a rather narrow range. Complexes **1** and **2** correspond to the two possible configurations below.

There are two kinds of sodium cation, two of them being bonded to the Schiff base only, and the other two completing their co-ordination sphere with a dme molecule. All of them are five-co-ordinated. The geometries of salphen and salen are identified by the parameters given in Table 2. They are in good agreement with those generally observed in Schiff-base complexes when the metal is not coplanar with the N_2O_2

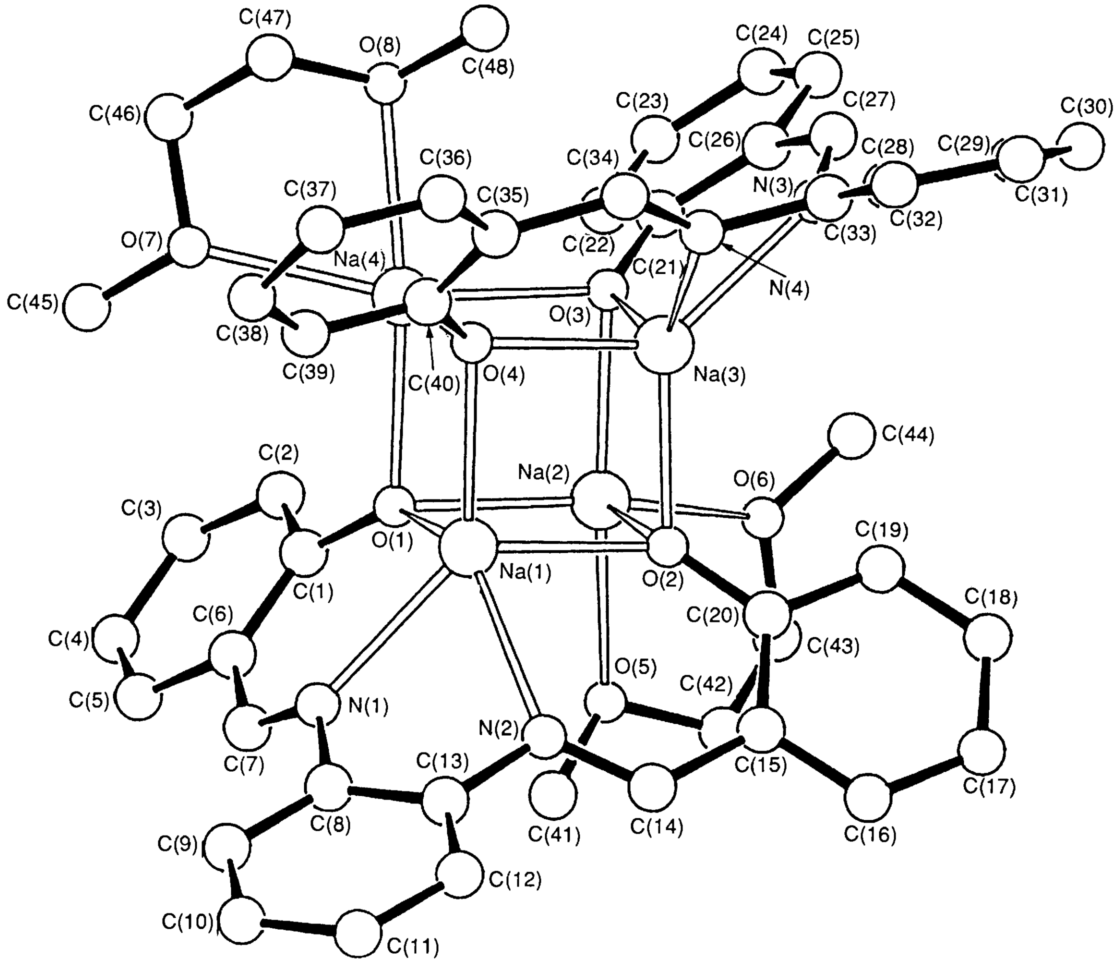
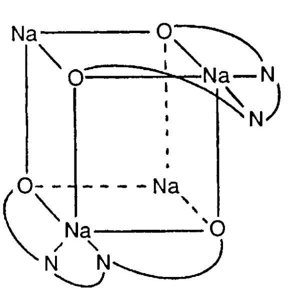
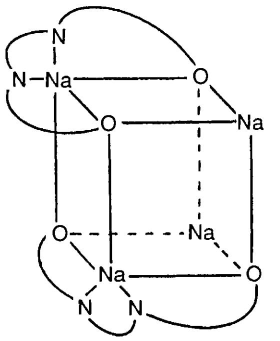


Fig. 1 A SCHAKAL[5] view of complex **1**





core.[6,7] The torsion angles around the ethylenic bridge indicate the presence of enantiomeric λ and δ conformations for the two chelate five-membered rings in molecule A of complex **2**. The conformations of the two rings in molecule B are λ,λ. Molecules enantiomeric with these are present in the structure generated by a centre of symmetry.

Experimental

All the reactions were carried out under an atmosphere of purified nitrogen. Solvents were dried and distilled before use by standard methods. The ^{1}H NMR spectra were recorded on a Bruker AC200 instrument. The syntheses of H_2salphen and H_2salen were carried out by condensing salicylaldehyde and the corresponding diamine in EtOH and then refluxing the suspension formed for 1 h.

Preparation of **1**.—Sodium hydride (1.67 g, 69.52 mmol) was added slowly to a thf (300 cm^3) solution of H_2salphen (10.0 g, 31.6 mmol). A significant evolution of H_2 was observed, then the solution was refluxed for 30 min. The excess of NaH was filtered off and the solution cooled to -10 °C overnight. The resulting microcrystalline solid was crystallized from dme (85%) [Found: C, 63.95; H, 5.75; N, 6.30. Na_2(salphen)(dme), $C_{24}H_{24}N_2Na_2O_4$ requires C, 64.00; H, 5.35; N, 6.20%]. ^{1}H NMR (CD_3CN, 200 MHz): δ 8.34 (s, 2 H, CH), 7.21 (m, 6 H, Ph), 7.05 (m, 2 H, Ph), 6.50 (d, 2 H, Ph), 6.34 (m, 2 H, Ph), 3.51 (s, 4 H, dme), 3.34 (s, 6 H, dme). IR(Nujol): ν(CH=N): 1623s cm^{-1}.

Preparation of **2**.—Sodium hydride (2.0 g, 83.3 mmol) in slight excess was added slowly to a thf (300 cm^3) solution of H_2salen (10.0 g, 33.9 mmol). A significant evolution of H_2 was observed, then the solution was refluxed for 30 min. The excess of NaH was filtered off and the solution concentrated to 100 cm^3 and cooled to -10 °C. A yellow crystalline solid was isolated and crystallized from dme to give crystals suitable for X-ray analysis (80%) [Found: C, 59.90; H, 6.50; N, 7.10. Na_2(salen)(dme), $C_{20}H_{24}N_2Na_2O_4$ requires C, 59.70; H, 6.00; N, 6.95%]. ^{1}H NMR (CD_3CN, 200 MHz): δ 8.18 (s, 2 H, CH), 7.05 (m, 2 H, Ph), 6.83 (m, 2 H, Ph), 6.40 (m, 2 H, Ph), 6.27 (m, 2 H, Ph), 3.62 (m, 4 H, CH_2–CH_2), 3.51 (s, 4 H, dme), 3.34 (s, 6 H, dme). IR(Nujol): ν(CH=N) 1641s cm^{-1}.

X-Ray Crystallography.—Crystal structure determination of complexes **1** *and* **2**. Crystal data and details associated with the data collection and refinement are quoted in Table 3. The crystal quality was tested by ψ scans showing that crystal absorption effects could be neglected. The structures were

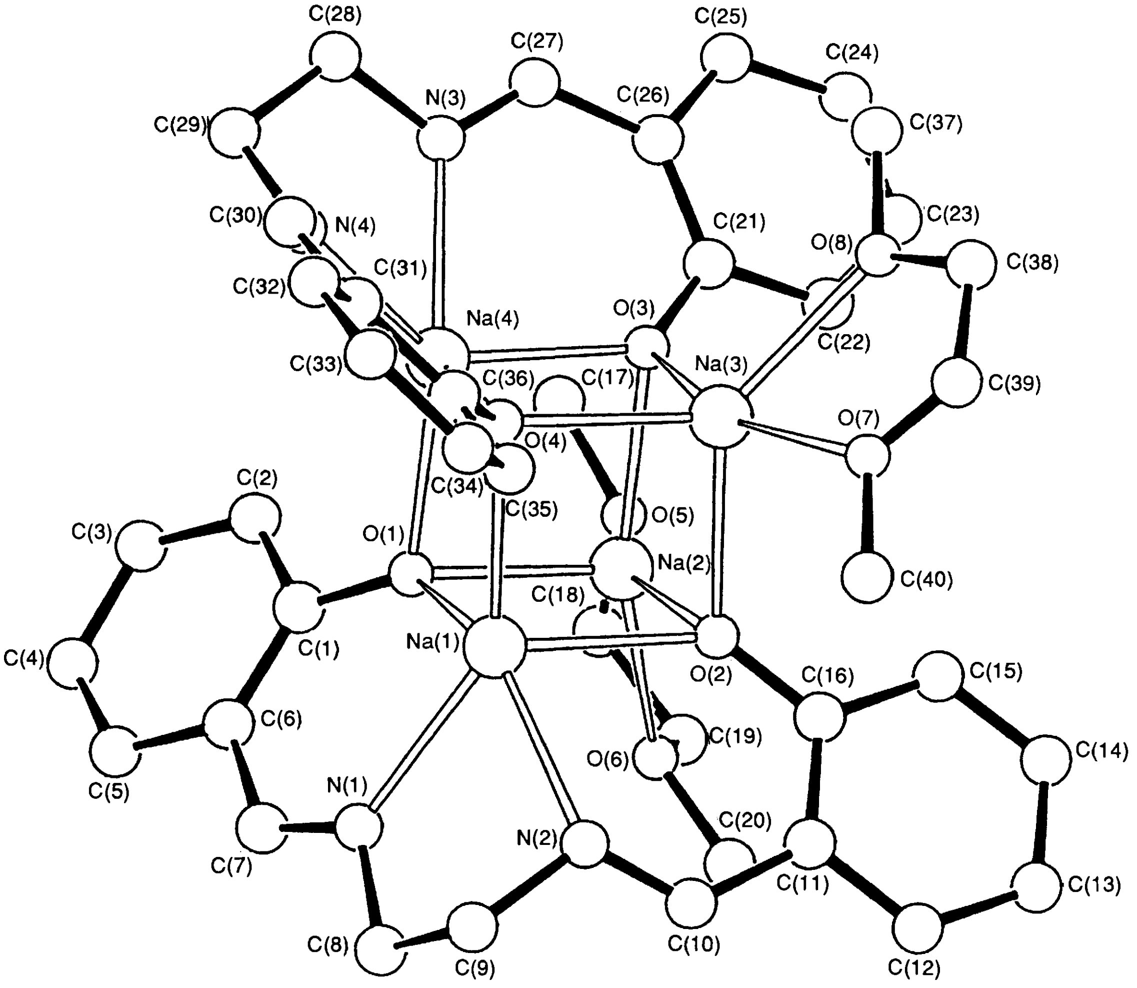


Fig. 2 A SCHAKAL[5] view of complex **2** (molecule A)

Table 3 Experimental data for the X-ray diffraction studies for complexes **1** and **2**[a]

	1	**2**
Formula	$C_{48}H_{48}N_4Na_4O_8$	$C_{40}H_{48}N_4Na_4O_8$
M	900.9	804.8
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/n$
Cell parameters at 295 K[b]		
a/Å	12.045(1)	20.493(2)
b/Å	25.851(2)	21.682(2)
c/Å	16.467(1)	20.538(2)
α,γ/°	90	90
β/°	107.61(1)	110.53(1)
U/Å^3	4887.1(7)	8546.1(15)
Z	4	8
D_c/g cm^{-3}	1.224	1.251
F(000)	1888	3392
Linear absorption coefficient/cm^{-1}	9.58	10.33
Crystal dimensions/mm	0.20 × 0.28 × 0.30	0.25 × 0.30 × 0.36
Unique total data	7270	11 279
Unique observed data	2118	5054
$R = \Sigma\lvert\Delta F\rvert/\Sigma\lvert F_o\rvert$	0.079	0.071

[a] Details common to both complexes: Siemens AED diffractometer; θ–2θ scan type; nickel-filtered Cu-Kα radiation (λ = 1.541 78 Å); 2θ range 6–120°; criterion for observation $I > 2\sigma(I)$. [b] Unit-cell parameters were obtained by least-squares analysis of the setting angles of 25 carefully centred reflections chosen from diverse regions of reciprocal space.

solved using SHELX 86.[8] For complex **1** refinement was first carried out isotropically, then anisotropically for the sodium, oxygen and nitrogen atoms only owing to the very low percentage of observed reflections. For complex **2** refinement was first isotropic, then anisotropic for all the non-hydrogen atoms. For both complexes all hydrogen atoms were put in calculated positions and introduced into the refinement as fixed contributors with isotropic U values fixed at 0.08 Å^2. During the refinements all the aromatic rings were constrained to be regular hexagons (C–C 1.395 Å). Final atomic coordinates are listed in Tables 4 and 5.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates, thermal parameters and remaining bond distances and angles.

Acknowledgements

We thank the Fonds National Suisse de la Recherche Scientifique (Grant No. 20–28470.90) for financial support.

References

1 M. Calligaris and L. Randaccio, in *Comprehensive Coordination Chemistry*, eds. G. Wilkinson, R. D. Gillard and J. A. McCleverty, Pergamon, Oxford, 1987, vol. 2, ch. 20.1, p. 715 and refs. therein.
2 R. H. Holm, G. W. Everett, jun., and A. Chakravorty, *Prog. Inorg. Chem.*, 1966, **7**, 83.
3 M. Mazzanti, C. Floriani, A. Chiesi-Villa and C. Guastini, *Inorg. Chem.*, 1986, **25**, 4158; M. Mazzanti, S. Gambarotta, C. Floriani, A. Chiesi-Villa and C. Guastini, *Inorg. Chem.*, 1986, **25**, 2308; F. Corazza, E. Solari, C. Floriani, A. Chiesi-Villa and C. Guastini, *J. Chem. Soc., Dalton Trans.*, 1990, 1335 and refs. therein.

Table 4 Atomic coordinates (× 10^4) for complex **1**

Atom	*X*/*a*	*Y*/*b*	*Z*/*c*
Na(1)	3 687(3)	6 575(1)	8 540(2)
Na(2)	1 962(4)	5 674(1)	7 513(3)
Na(3)	1 048(4)	6 784(1)	7 825(3)
Na(4)	2 491(4)	6 739(1)	6 513(2)
O(1)	3 650(6)	6 096(3)	7 390(4)
O(2)	2 103(6)	6 184(3)	8 724(4)
O(3)	794(6)	6 302(3)	6 615(4)
O(4)	2 602(6)	7 246(3)	7 731(4)
O(5)	2 912(9)	4 930(4)	8 218(7)
O(6)	599(9)	4 975(3)	7 347(7)
O(7)	3 802(8)	7 085(4)	5 832(6)
O(8)	1 464(8)	7 341(3)	5 493(5)
N(1)	5 644(8)	6 276(3)	8 918(6)
N(2)	4 336(8)	6 333(3)	9 996(6)
N(3)	−950(8)	6 841(3)	7 250(6)
N(4)	462(8)	7 621(3)	8 071(5)
C(1)	4 458(7)	5 837(3)	7 144(6)
C(2)	4 097(7)	5 613(3)	6 336(6)
C(3)	4 894(7)	5 345(3)	6 031(6)
C(4)	6 052(7)	5 301(3)	6 534(6)
C(5)	6 413(7)	5 524(3)	7 342(6)
C(6)	5 616(7)	5 793(3)	7 648(6)
C(7)	6 125(11)	5 981(5)	8 508(8)
C(8)	6 216(7)	6 422(3)	9 782(4)
C(9)	7 392(7)	6 555(3)	10 072(4)
C(10)	7 881(7)	6 731(3)	10 905(4)
C(11)	7 194(7)	6 772(3)	11 448(4)
C(12)	6 017(7)	6 639(3)	11 159(4)
C(13)	5 528(7)	6 463(3)	10 325(4)
C(14)	3 812(11)	6 089(5)	10 459(8)
C(15)	2 593(6)	5 950(3)	10 198(6)
C(16)	2 221(6)	5 757(3)	10 863(6)
C(17)	1 054(6)	5 625(3)	10 718(6)
C(18)	259(6)	5 686(3)	9 909(6)
C(19)	631(6)	5 879(3)	9 244(6)
C(20)	1 798(6)	6 011(3)	9 388(6)
C(21)	−169(6)	6 155(3)	6 002(5)
C(22)	8(6)	5 822(3)	5 386(5)
C(23)	−943(6)	5 625(3)	4 748(5)
C(24)	−2 073(6)	5 761(3)	4 727(5)
C(25)	−2 250(6)	6 094(3)	5 344(5)
C(26)	−1 298(6)	6 291(3)	5 981(5)
C(27)	−1 631(11)	6 652(5)	6 561(8)
C(28)	−1 382(8)	7 172(3)	7 772(5)
C(29)	−2 467(8)	7 103(3)	7 899(5)
C(30)	−2 834(8)	7 442(3)	8 426(5)
C(31)	−2 116(8)	7 850(3)	8 826(5)
C(32)	−1 032(8)	7 919(3)	8 699(5)
C(33)	−665(8)	7 580(3)	8 172(5)
C(34)	975(11)	8 054(5)	8 058(8)
C(35)	2 165(5)	8 124(3)	8 013(5)
C(36)	2 562(5)	8 632(3)	8 167(5)
C(37)	3 689(5)	8 759(3)	8 170(5)
C(38)	4 420(5)	8 376(3)	8 018(5)
C(39)	4 023(5)	7 868(3)	7 864(5)
C(40)	2 896(5)	7 742(3)	7 861(5)
C(41)	3 954(18)	5 002(7)	8 868(12)
C(42)	2 157(21)	4 643(9)	8 490(15)
C(43)	1 079(16)	4 532(7)	7 793(11)
C(44)	−597(16)	4 952(7)	6 959(11)
C(45)	4 815(17)	6 876(7)	5 719(11)
C(46)	3 225(14)	7 413(6)	5 134(10)
C(47)	2 264(14)	7 684(6)	5 321(10)
C(48)	523(14)	7 585(6)	5 669(10)

Table 5 Atomic coordinates (× 10^4) for complex **2**

	Molecule A			Molecule B		
Atom	*X*/*a*	*Y*/*b*	*Z*/*c*	*X*/*a*	*Y*/*b*	*Z*/*c*
Na(1)	6963(2)	1466(1)	1550(2)	1842(2)	1536(2)	3529(2)
Na(2)	7691(2)	2414(2)	2801(2)	1198(2)	2547(1)	2301(2)
Na(3)	6340(2)	1523(2)	2810(2)	2548(2)	1649(2)	2309(2)
Na(4)	6124(2)	2673(2)	1776(2)	2727(2)	2706(2)	3419(2)
O(1)	7163(3)	2501(3)	1616(3)	1650(3)	2567(3)	3511(3)
O(2)	7436(3)	1315(3)	2758(3)	1434(3)	1454(3)	2330(3)
O(3)	6620(3)	2600(2)	2955(3)	2310(3)	2742(3)	2227(3)
O(4)	5820(3)	1660(3)	1602(3)	2976(3)	1679(3)	3514(3)
O(5)	8219(4)	3402(3)	3351(4)	677(4)	3547(4)	1843(5)
O(6)	8894(4)	2407(4)	3076(5)	−31(4)	2536(4)	1955(5)
O(7)	6116(5)	461(4)	3088(5)	2756(5)	673(4)	1851(5)
O(8)	5807(7)	1467(4)	3628(7)	3573(5)	1683(4)	2046(6)
N(1)	7709(3)	1572(3)	891(4)	1108(4)	1611(3)	4195(4)
N(2)	7635(4)	565(3)	1656(5)	1140(5)	651(3)	3363(5)
N(3)	5490(5)	3450(4)	2138(6)	3276(5)	3578(4)	3229(5)
N(4)	5008(4)	2733(4)	965(5)	3885(5)	2698(4)	4193(5)
C(1)	7349(3)	2889(2)	1196(3)	1451(3)	2935(2)	3932(3)
C(2)	7227(3)	3516(2)	1258(3)	1537(3)	3567(2)	3863(3)
C(3)	7417(3)	3946(2)	852(3)	1327(3)	3984(2)	4268(3)
C(4)	7730(3)	3750(2)	383(3)	1031(3)	3770(2)	4742(3)
C(5)	7852(3)	3123(2)	321(3)	946(3)	3138(2)	4811(3)
C(6)	7662(3)	2693(2)	728(3)	1156(3)	2720(2)	4406(3)
C(7)	7828(4)	2048(5)	600(5)	1005(4)	2080(4)	4515(5)
C(8)	8013(6)	994(5)	748(6)	834(5)	1023(4)	4334(6)
C(9)	7685(6)	442(5)	963(6)	1093(6)	499(4)	4051(6)
C(10)	8114(5)	341(4)	2178(6)	658(6)	484(4)	2810(7)
C(11)	8220(4)	465(3)	2920(3)	560(3)	633(3)	2090(3)
C(12)	8726(4)	88(3)	3378(3)	107(3)	275(3)	1591(4)
C(13)	8899(4)	159(3)	4094(3)	9(3)	349(3)	889(4)
C(14)	8566(4)	608(3)	4351(3)	403(3)	781(3)	684(4)
C(15)	8060(4)	985(3)	3893(3)	896(3)	1139(3)	1183(4)
C(16)	7887(3)	914(3)	3177(3)	994(3)	1065(3)	1886(4)
C(17)	7833(7)	3966(5)	3242(7)	1031(6)	4101(5)	1887(6)
C(18)	8875(7)	3467(6)	3290(7)	35(7)	3607(6)	1972(8)

Table 5 *continued*

	Molecule A			Molecule B		
Atom	*X*/*a*	*Y*/*b*	*Z*/*c*	*X*/*a*	*Y*/*b*	*Z*/*c*
C(19)	9280(6)	2897(7)	3498(6)	−388(6)	3072(8)	1692(7)
C(20)	9273(6)	1852(7)	3242(10)	−431(7)	2008(8)	1702(11)
C(21)	6612(3)	2957(3)	3484(3)	2419(3)	3138(3)	1767(3)
C(22)	7113(3)	2849(3)	4138(3)	2058(3)	3020(3)	1064(3)
C(23)	7122(3)	3208(3)	4705(3)	2131(3)	3417(3)	559(3)
C(24)	6631(3)	3676(3)	4618(3)	2563(3)	3932(3)	757(3)
C(25)	6130(3)	3784(3)	3964(3)	2924(3)	4050(3)	1459(3)
C(26)	6120(3)	3425(3)	3397(3)	2852(3)	3653(3)	1964(3)
C(27)	5552(6)	3594(4)	2747(7)	3295(6)	3825(5)	2672(7)
C(28)	4836(6)	3636(5)	1590(7)	3793(10)	3770(7)	3900(8)
C(29)	4781(6)	3379(5)	893(7)	4230(8)	3301(7)	4264(8)
C(30)	4580(5)	2323(6)	656(6)	4251(5)	2252(5)	4528(5)
C(31)	4693(3)	1655(3)	701(3)	4047(3)	1602(3)	4474(3)
C(32)	4150(3)	1306(3)	249(3)	4522(3)	1226(3)	4967(3)
C(33)	4203(3)	665(2)	245(3)	4409(3)	592(3)	4963(3)
C(34)	4798(3)	373(3)	692(3)	3822(3)	333(3)	4465(3)
C(35)	5341(3)	722(3)	1143(3)	3347(3)	709(3)	3972(3)
C(36)	5288(3)	1363(7)	1148(3)	3459(3)	1344(3)	3977(3)
C(37)	5483(9)	1953(7)	3842(11)	3930(8)	2227(7)	2141(10)
C(38)	5663(12)	890(8)	3854(10)	3797(9)	1160(8)	1906(10)
C(39)	5910(19)	416(8)	3550(16)	3391(11)	658(7)	1793(11)
C(40)	6391(7)	−53(6)	2845(7)	2392(8)	147(6)	1768(11)

4 D. E. Fenton, in *Comprehensive Coordination Chemistry*, eds. G. Wilkinson, R. D. Gillard and J. A. McCleverty, Pergamon, Oxford, 1987, vol. 3, ch. 23.

5 E. Keller, SCHAKAL 88B/V16, A Fortran Program for Graphical Representation of Molecular and Crystallographic Models, University of Freiburg, 1990.

6 M. D. Hobday and T. D. Smith, *Coord. Chem. Rev.*, 1972, **9**, 311.

7 M. Calligaris, G. Nardin and L. Randaccio, *Coord. Chem. Rev.*, 1972, **7**, 385.

8 G. M. Sheldrick, SHELX 86, Program for crystal structure solution, University of Göttingen, 1986.

Received 15*th April* 1991; *Paper* 1/01739J