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Structure of a Zinc(II) Complex with a Non-Symmetrical Tetradentate Schiff Base

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Abstract. {*N*-[2-(2-Aminoethylamino)ethyl]salicylideneaminato-*O*,*N*,*N*′,*N*″}aquazinc(II) bromide, $[Zn(C_{11}H_{16}N_3O)(H_2O)]Br$, $M_r = 369{\cdot}57$, monoclinic, $P2_1/c$, $a = 9{\cdot}129\ (1)$, $b = 14{\cdot}207\ (1)$, $c = 11{\cdot}003\ (1)$ Å, $\beta = 102{\cdot}36\ (1)^\circ$, $V = 1393{\cdot}9\ (2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}77\ (1)$, $D_x = 1{\cdot}76$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 4{\cdot}6$ mm^{-1}, $F(000) = 744$, $T = 295$ K, $R = 0{\cdot}030$ for 1995 unique observed reflections. The crystal comprises a cationic Zn^{II} complex which includes the saden ligand (saden = {*N*-[2-(2-aminoethylamino)ethyl]salicylideneaminato-*O*,*N*,*N*′,*N*″}) and a water molecule. The Zn atom is surrounded by three N and two O atoms (one from H_2O) with a distorted trigonal bipyramidal arrangement. Intermolecular hydrogen bridges of the O(water)···O type join the molecules in dimers. Other than this, there is one Br···HO(water) and two Br···HN intermolecular distances, which correspond to hydrogen bonds much weaker than the O···O ones, in agreement with infrared spectra ($\nu_{Br\cdots DO} = 2381$ cm^{-1}, $\nu_{Br\cdots DN} =$ 2347 and 2326 cm^{-1}).

Experimental. To an aqueous solution of $ZnBr_2$ (2·25 g, 0·01 ml) an equivalent amount of the Schiff base in methanol solution was slowly added. The resulting mixture was neutralized by 0·84 g (0·01 mol) $NaHCO_3$, filtered and allowed to stand in air. Small yellowish crystals were obtained over a few days. The yield was 1·3 g (35%). Density was determined by flotation in a methylene iodide/toluene mixture. Parameters describing the data

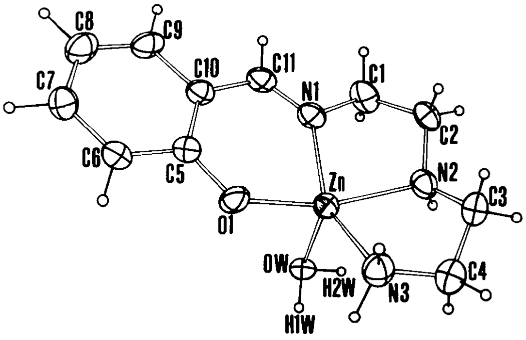

Fig. 1. View of the complex cation with atom numbering. Thermal ellipsoids are scaled to 50% probability.

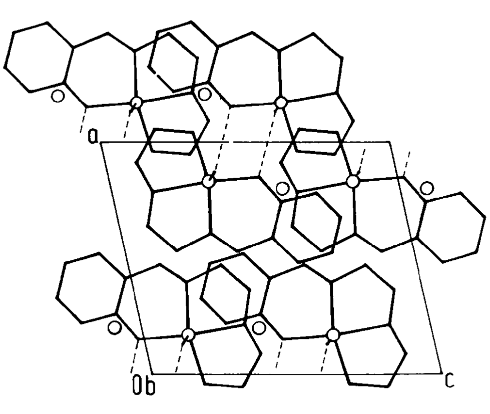

Fig. 2. Packing scheme. The intermolecular O···O hydrogen bonds are indicated by dashed lines.

Table 1. *Data collection and structure refinement parameters*

Crystal dimensions	0·36 × 0·28 × 0·20 mm
Diffractometer and radiation used	Enraf–Nonius CAD-4, graphite monochromator, λ(Mo $K\alpha$) = 0·71073 Å
Scan technique	$\omega/2\theta$
No. and θ range of reflections for lattice parameter refinement	23; 22·59–24·72°
Range of h, k, l	$-10\rightarrow10$, $0\rightarrow16$, $-13\rightarrow13$
Maximum value of $(\sin\theta)/\lambda$	0·595 Å^{-1}
Standard reflections	$11\bar{1}$, $10\bar{2}$
Interval in which standard reflections monitored; intensity fluctuation	120 min; −2·1%
Total number of reflections measured; 2θ range	2283; $2\theta \leq 50°$
Value of R_{int}	0·019
No. of unique observed reflections	1995
Criterion for observed reflections	$I > 1{\cdot}96\sigma(I)$
Function minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Weighting scheme	$w = 1/[\sigma^2(F_o) + 0{\cdot}0009F_o^2]$
Parameters refined	235
Value of R	0·030
Value of wR	0·030
Value of S	0·92
Ratio of max. least-squares shift to e.s.d. (Δ/σ) in the last cycle	0·003
Max. and min. heights in final $\Delta\rho$ map	0·39, −0·80 e Å^{-3}
Source of atomic scattering factors	*SHELX*76 and *International Tables for X-ray Crystallography* (1974, Vol. IV)
Programs used	*SDP* (B. A. Frenz & Associates, Inc., 1985), *SHELX*76 (Sheldrick, 1976)
Computer used	DEC PDP11/73

Table 2. *Atomic coordinates* (× 10⁴) *for non*-H *atoms and equivalent isotropic thermal parameters* (× 10⁴)

$U_{eq} = (1/3)[U_{22} + 1/\sin^2\beta(U_{11} + U_{33} + 2U_{13}\cos\beta)]$.

	x	y	z	U_{eq} (Å^2)
Br	2004·6 (4)	8250·6 (3)	9050·4 (4)	410 (1)
Zn	1705·9 (4)	4417·2 (3)	1558·9 (3)	282 (1)
O1	1520 (2)	4020 (2)	−211 (2)	387 (7)
O*W*	1309 (3)	5822 (2)	1287 (3)	312 (7)
N1	3951 (3)	4165 (2)	1897 (2)	307 (8)
N2	2148 (4)	4351 (2)	3576 (3)	364 (9)
N3	−396 (4)	3944 (3)	1729 (3)	413 (10)
C1	4692 (4)	4290 (3)	3206 (3)	404 (11)
C2	3639 (4)	3942 (3)	4017 (3)	351 (10)
C3	895 (5)	3827 (3)	3911 (4)	470 (13)
C4	−548 (5)	4077 (4)	3019 (4)	509 (14)
C5	2593 (4)	3789 (2)	−798 (3)	295 (9)
C6	2206 (4)	3554 (3)	−2068 (3)	338 (10)
C7	3263 (4)	3298 (3)	−2723 (4)	384 (12)
C8	4773 (5)	3282 (3)	−2161 (4)	428 (12)
C9	5193 (4)	3498 (2)	−921 (4)	360 (11)
C10	4133 (4)	3744 (2)	−207 (3)	289 (9)
C11	4720 (4)	3915 (3)	1107 (3)	321 (10)

Table 3. *Bond lengths* (Å) *and angles* (°)

Zn—O1	2·000 (2)	N3—C4	1·468 (6)
Zn—O*W*	2·039 (3)	C1—C2	1·527 (6)
Zn—N1	2·035 (3)	C3—C4	1·507 (6)
Zn—N2	2·171 (3)	C5—C6	1·406 (5)
Zn—N3	2·079 (4)	C5—C10	1·418 (5)
O1—C5	1·325 (4)	C6—C7	1·371 (6)
N1—C1	1·465 (4)	C7—C8	1·385 (6)
N1—C11	1·279 (5)	C8—C9	1·371 (6)
N2—C2	1·464 (5)	C9—C10	1·415 (6)
N2—C3	1·476 (6)	C10—C11	1·451 (4)
N2—Zn—N3	82·4 (1)	Zn—N3—C4	109·0 (3)
N1—Zn—N3	147·1 (1)	N1—C1—C2	108·6 (3)
N1—Zn—N2	81·1 (1)	N2—C2—C1	109·6 (3)
O*W*—Zn—N3	101·0 (1)	N2—C3—C4	109·6 (4)
O*W*—Zn—N2	100·4 (1)	N3—C4—C3	110·4 (4)
O*W*—Zn—N1	109·9 (1)	O1—C5—C10	123·5 (3)
O1—Zn—N3	96·3 (1)	O1—C5—C6	119·2 (3)
O1—Zn—N2	160·4 (1)	C6—C5—C10	117·3 (3)
O1—Zn—N1	90·0 (1)	C5—C6—C7	122·0 (3)
O1—Zn—O*W*	99·1 (1)	C6—C7—C8	121·0 (4)
Zn—O1—C5	128·9 (2)	C7—C8—C9	118·7 (4)
Zn—N1—C11	127·1 (2)	C8—C9—C10	122·0 (4)
Zn—N1—C1	113·2 (2)	C5—C10—C9	119·0 (3)
C1—N1—C11	119·8 (3)	C9—C10—C11	116·4 (3)
Zn—N2—C3	107·0 (2)	C5—C10—C11	124·6 (3)
Zn—N2—C2	108·2 (2)	N1—C11—C10	125·8 (3)
C2—N2—C3	115·6 (3)		
Intermolecular hydrogen bonds			
O*W*···O1^{i}	2·613 (4)	O*W*—H1*W*···O1^{i}	164 (4)
Br···O*W*^{ii}	3·249 (3)	Br···H2*W*^{ii}—O*W*^{ii}	167 (4)
Br···N2^{ii}	3·454 (3)	Br···HN2^{ii}—N2^{ii}	164 (3)
Br···N3^{iii}	3·475 (4)	Br···H2N3^{iii}—N3^{iii}	167 (5)

Symmetry code: (i) $-x, -y+1, -z$; (ii) $x, \frac{3}{2}-y, z-\frac{1}{2}$; (iii) $-x, 1-y, 1-z$.

collection and structure refinement are listed in Table 1. The structure was solved from Patterson and $\Delta\rho$ maps and refined with anisotropic thermal parameters by block-diagonal least squares with the parameters partitioned between three blocks: (i) scale factor, positional and thermal parameters from Br to N3 (see Table 2) and corresponding H atoms; (ii) parameters from C1 to C5 with H atoms; (iii) parameters from C6 to C11 with H atoms. Water H atoms were localized in a $\Delta\rho$ map, the other H atoms were introduced in calculated positions. All H atoms were refined isotropically. An empirical absorption correction was applied (Walker & Stuart, 1983), the absorption factors varied from 1·360 to 1·756. Atomic parameters are given in Table 2,* bond lengths and angles in Table 3. The complex cation is drawn in Fig. 1 and the crystal packing in Fig. 2.

Related literature. Crystal structure information is available for [Ni(saden)]B$(C_6H_5)_4$ (Rotondo, Cusmano Priolo, Romeo, Bruno & Bombieri, 1983),

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* Lists of structure factors, anisotropical thermal parameters, H-atom positions and isotropic thermal parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54074 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

[Ni(saden)]ClO_4 (Podlahová, Knížek, Loub & Hašek, 1988), [Ni(saden)]NCS (Loub, Podlahová, Kopf & Weiss, 1989), [Pd(saden)]ClO_4 (Kratochvíl, Nováková, Haber, Ondráček & Hájek, 1989), [Cu(saden)H_2O]PF_6 (Cusmano Priolo, Rotondo, Rizzardi, Bruno & Bombieri, 1983) and [Cd(saden)C_2H_5OH]ClO_4 (Loub, Podlahová, Haber, Kopf & Weiss, 1990).

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Structure of Carbonyl(2-quinolinecarboxylato-$\kappa N,\kappa O$)-(triphenylphosphite-κP)rhodium(I)

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Abstract. $[Rh(C_{10}H_6NO_2)(CO)\{P(C_6H_5O)_3\}]$, M_r = 613·4, monoclinic, $P2_1/c$, a = 11·727 (6), b = 11·380 (5), c = 19·866 (3) Å, β = 102·08 (2)°, V = 2592·5 Å^3, Z = 4, D_m = 1·53, D_x = 1·57 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 7·70 cm^{-1}, F(000) = 1240, final R = 0·042 for 2796 observed reflections. The structure indicates that the complex has square-planar geometry about the Rh atom and that the carbonyl group *trans* to the N atom of the 2-carboxyquinolinato ligand was substituted by the triphenylphosphite ligand. The N atom of the chelate ring therefore has a larger thermodynamic *trans* influence than the O atom. The π-acceptor properties of triphenylphosphite resulted in a relatively strong bond between Rh and P [bond distance 2·172 (2) Å] as compared to the Rh—P bond [bond distance 2·258 (2) Å] in the corresponding phosphine complex.

Experimental. An equivalent amount of triphenylphosphite (0·23 g dissolved in 1 cm^3 acetone) was added dropwise to a suspension of 0·25 g [Rh(quin)-$(CO)_2$] in 2 cm^3 acetone, where quin is the 2-quinolinecarboxylato ligand (Graham, Lamprecht, Potgieter, Roodt & Leipoldt, 1990). The resulting yellow precipitate was recrystallized from acetone to yield well formed crystals of the title compound, suitable for X-ray analysis. The density was determined by flotation in sodium iodide solution. A crystal of dimensions 0·20 × 0·15 × 0·35 mm was used for data collection on an Enraf–Nonius CAD-4F diffractometer with graphite-monochromated Mo $K\alpha$ radiation. The $\omega/2\theta$-scan technique, with variable scan width $\Delta\omega$ = (0·53 + 0·34tanθ)°, scan speed 3·30° min^{-1} in ω and a maximum scan time of 50 s per reflection, was used. The unit-cell parameters were determined from least-squares refinement of 25 reflections with $7 < \theta < 16°$, with total measuring range $3 < \theta < 25°$. Empirical absorption corrections (North, Phillips & Matthews, 1968) with a minimum correction factor 0·90 and maximum 0·99 were used. The mean intensity of three standard reflections was measured over 3600 s of X-ray exposure time, and varied from the initial value by −2·0%. All possible reflections with $(\sin\theta)/\lambda < 0·71$ Å^{-1} in the index range $0 < h < 13$, $0 < k < 13$, $-23 < l < 23$, gave 3947 unique with 2796 considered as observed [$I > 3·0\sigma(I)$]. The structure was solved by the heavy-atom method using *SHELXS86* (Sheldrick, 1990) and was subjected to anisotropic full-matrix least-squares refinement on F using *SHELX76* (Sheldrick, 1976). The H-atom positions were calculated riding on the adjacent C atoms assuming C—H = 1·08 Å, and were refined with an overall temperature factor. Neutral-atom scattering factors (Cromer & Mann, 1968) and

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