

O(2)	0.4035 (4)	-0.1162 (2)	0.0196 (3)	0.0326 (7)
O(3)	0.3162 (3)	0.0490 (2)	0.3418 (2)	0.0211 (6)
O(4)	-0.0307 (3)	0.0856 (2)	0.3246 (2)	0.0200 (6)
O(5)	0.1896 (3)	0.2043 (2)	0.2274 (2)	0.0221 (6)
C(1)	0.0936 (5)	0.1080 (2)	-0.1198 (3)	0.0191 (8)
C(2)	0.2587 (5)	-0.0742 (2)	0.0148 (3)	0.0209 (9)
C(3)	0.5099 (5)	0.0590 (3)	0.3135 (4)	0.0256 (9)
C(4)	0.0116 (5)	0.1333 (3)	0.4665 (4)	0.0254 (9)
C(5)	0.0547 (5)	0.2776 (2)	0.1466 (4)	0.0277 (10)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Mo—P(1)	2.4256 (8)	O(1)—C(1)	1.145 (4)
Mo—C(1)	2.041 (3)	O(2)—C(2)	1.149 (4)
Mo—C(2)	2.037 (4)	O(3)—C(3)	1.451 (4)
P(1)—O(3)	1.606 (2)	O(4)—C(4)	1.458 (4)
P(1)—O(4)	1.595 (2)	O(5)—C(5)	1.445 (4)
P(1)—O(5)	1.611 (2)		
P(1)—Mo—C(1)	93.30 (9)	O(3)—P(1)—O(5)	97.0 (1)
P(1)—Mo—C(2)	93.12 (9)	O(4)—P(1)—O(5)	104.6 (1)
C(1)—Mo—C(2)	89.0 (1)	P(1)—O(3)—C(3)	120.3 (2)
Mo—P(1)—O(3)	120.52 (9)	P(1)—O(4)—C(4)	121.3 (2)
Mo—P(1)—O(4)	111.16 (9)	P(1)—O(5)—C(5)	119.9 (2)
Mo—P(1)—O(5)	121.02 (9)	Mo—C(1)—O(1)	179.6 (3)
O(3)—P(1)—O(4)	99.3 (1)	Mo—C(2)—O(2)	178.2 (3)

The diffractometer was equipped with an Oxford Systems low-temperature attachment (Cosier & Glazer, 1986). As there were no identifiable faces, the data were corrected for absorption using  $\psi$  scans. All non-H atoms were refined anisotropically. H atoms were included in fixed calculated positions assuming staggering across O—CH<sub>3</sub> bonds, with a C—H distance of 0.96 Å.  $U(\text{H})$  was set equal to  $1.2U_{\text{eq}}(\text{C})$  and left unaltered during subsequent refinement.

Data collection: *MSCI/AFSC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCI/AFSC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We thank the University of Southampton for support and the EPSRC for a grant to purchase the diffractometer.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1224). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Andrews, G. T., Colquhoun, I. J., McFarlane, W. & Grim, S. O. (1982). *J. Chem. Soc. Dalton Trans.* pp. 2353–2358.
- Aroney, M. J., Davies, M. S., Hambley, T. W. & Picrens, R. K. (1994). *J. Chem. Soc. Dalton Trans.* pp. 91–96.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Greene, N., Taylor, H., Kee, T. P. & Thornton-Pett, M. (1993). *J. Chem. Soc. Dalton Trans.* pp. 821–825.
- Molecular Structure Corporation (1988). *MSCI/AFSC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1992). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.

*Acta Cryst.* (1996). **C52**, 799–801

## [Pt(PMe<sub>3</sub>)<sub>2</sub>(dppm)]Cl<sub>2</sub>·2H<sub>2</sub>O [dppm = Bis-(diphenylphosphino)methane]

ANDREW F. CHIFFEY, JOHN EVANS, WILLIAM LEVASON AND GILLIAN REID

Department of Chemistry, University of Southampton, Highfield, Southampton SO17 1BJ, England. E-mail: gr@soton.ac.uk

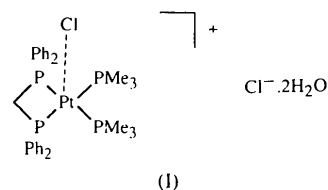
(Received 21 August 1995; accepted 23 October 1995)

## Abstract

The crystal structure of [bis(diphenylphosphino)methane]bis(trimethylphosphine)platinum(II) chloride dihydrate, [Pt(C<sub>3</sub>H<sub>9</sub>P)<sub>2</sub>(C<sub>25</sub>H<sub>22</sub>P<sub>2</sub>)]Cl<sub>2</sub>·2H<sub>2</sub>O, contains [Pt(PMe<sub>3</sub>)<sub>2</sub>(dppm)]<sup>2+</sup> cations with the Pt<sup>II</sup> ion bound to a distorted square-planar arrangement of one chelating dppm ligand and two PMe<sub>3</sub> ligands [Pt—P(dppm) 2.317 (6), 2.352 (7) Å; Pt—P(PMe<sub>3</sub>) 2.331 (7), 2.345 (7) Å]. A weakly interacting Cl<sup>-</sup> ion occupies an apical coordination site [Pt···Cl 3.175 (7) Å] and a discrete Cl<sup>-</sup> anion is present to balance the charges. Two H<sub>2</sub>O molecules are also present in the asymmetric unit.

## Comment

The title complex, (I), was prepared by the reaction of [Pt(dppm)<sub>2</sub>]Cl<sub>2</sub> with PMe<sub>3</sub> in MeCN solution. The X-ray structure (Fig. 1) shows a slightly distorted square-planar arrangement of phosphorus donors from one chelating dppm ligand and two PMe<sub>3</sub> ligands around the Pt<sup>II</sup> centre.



The Pt—P distances involving the dppm ligand (average 2.335 Å) and the PMe<sub>3</sub> ligands (average 2.337 Å) are slightly longer than the distances found in similar structures involving P<sub>4</sub> coordination around a Pt<sup>II</sup> centre, e.g. [Pt{Ph<sub>2</sub>PCH<sub>2</sub>CH<sub>2</sub>P(Ph)CH<sub>2</sub>CH<sub>2</sub>P(Ph)CH<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>}] (BPh<sub>4</sub>)<sub>2</sub>·3CH<sub>2</sub>Cl<sub>2</sub> (average Pt—P 2.30 Å; Brüggeller, Nar & Messerschmidt, 1992) and [Pt(Ph<sub>2</sub>PCH<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>)(Ph<sub>2</sub>PNHPPH<sub>2</sub>)]Cl<sub>2</sub>·CH<sub>2</sub>Cl<sub>2</sub> (average Pt—P 2.31 Å; Bhattacharyya, Sheppard, Slawin, Williams & Woollins, 1993). The P(1)—Pt(1)—P(2) angle in the five-membered chelate ring is 70.3 (2)°, similar to the values observed in other structurally characterized examples of complexes incorporating chelating

dppm ligands, *e.g.* *cis*-[PtPh<sub>2</sub>(dppm)] (73.0°; Braterman, Cross, Manojlović-Muir, Muir & Young, 1975) and *cis*-[PdCl<sub>2</sub>(dppm)] [72.68 (2)°; Steffan & Palenik, 1976].

One of the Cl<sup>-</sup> anions in the title structure occupies an apical coordination site and weakly interacts with the Pt<sup>II</sup> ion [Pt···Cl(1) 3.175 (7) Å] creating an overall geometry at Pt<sup>II</sup> approaching square-based pyramidal.

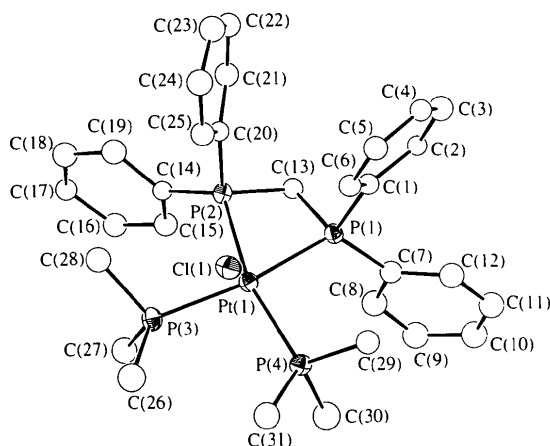


Fig. 1. View of the structure of [Pt(PMe<sub>3</sub>)<sub>2</sub>(dppm)]<sup>2+</sup> and its associated chloride ion, with the atom-numbering scheme. H atoms are omitted for clarity and displacement ellipsoids are drawn at the 40% probability level.

Two H<sub>2</sub>O molecules per cation were also identified. The water is thought to have originated from wet solvents and further evidence for its presence in the bulk sample was seen in the <sup>1</sup>H NMR spectrum and from the ν(OH) stretching vibration observed in the IR spectrum. The H atoms associated with the H<sub>2</sub>O solvent molecules were not located in the electron-density maps and therefore were not included in the model.

The Flack parameter (Flack, 1983; Bernardinelli & Flack, 1985) was refined in order to establish the correct absolute structure.

The closely related compounds [Pt(PR<sub>3</sub>)<sub>2</sub>(dppm)]<sup>2+</sup> and [PtCl(PR<sub>3</sub>)(dppm)]<sup>+</sup> (PR<sub>3</sub> = PBu<sub>3</sub>, PEt<sub>3</sub>, PMePh<sub>2</sub>, PPh<sub>3</sub>) have been synthesized previously and studied by <sup>31</sup>P{<sup>1</sup>H} NMR spectroscopy (Anderson & Lumetta, 1987). In CH<sub>2</sub>Cl<sub>2</sub> solution at 300 K the <sup>31</sup>P NMR spectrum of (1) was very broad, indicative of some fluxional process. At 245 K, however, a sharp second-order spectrum was observed similar to that observed by Anderson & Lumetta (1987). The <sup>195</sup>Pt NMR spectrum at 245 K showed the expected triplet of triplets, δ -4529 p.p.m. [<sup>1</sup>J(PtP)<sub>dppm</sub> 2050 Hz, <sup>1</sup>J(PtP)<sub>PMe<sub>3</sub></sub> 2610 Hz].

## Experimental

Single crystals were obtained by vapour diffusion of diethyl ether into a solution of the complex in MeCN. The selected crystal was coated with mineral oil and mounted on a glass fibre.

## Crystal data

[Pt(C<sub>3</sub>H<sub>9</sub>P)<sub>2</sub>(C<sub>25</sub>H<sub>22</sub>P<sub>2</sub>)]Cl<sub>2</sub>·2H<sub>2</sub>O

*M<sub>r</sub>* = 838.58

Monoclinic

*Cc*

*a* = 12.67 (1) Å

*b* = 15.73 (1) Å

*c* = 17.471 (9) Å

β = 102.76 (5)°

*V* = 3397 (4) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.640 Mg m<sup>-3</sup>

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 12.7–19.0°

μ = 4.546 mm<sup>-1</sup>

*T* = 150 K

Prism

0.30 × 0.30 × 0.25 mm

Colourless

## Data collection

AFC-7S diffractometer

ω/2θ scans

Absorption correction:

refined from Δ*F* (Walker & Stuart, 1983)

*T<sub>min</sub>* = 0.17, *T<sub>max</sub>* = 0.39

3271 measured reflections

3117 independent reflections

2647 observed reflections

[*I* > 3σ(*I*)]

*R<sub>int</sub>* = 0.159 (based on *F*<sup>2</sup>)

θ<sub>max</sub> = 25.07°

*h* = 0 → 15

*k* = 0 → 18

*l* = -20 → 20

3 standard reflections

monitored every 150

reflections

intensity decay: 0.42%

## Refinement

Refinement on *F*

*R* = 0.071

*wR* = 0.080

*S* = 4.28

2647 reflections

204 parameters

H-atom parameters *x<sub>y</sub>z*

calculated; *U*(H) = *U<sub>eq</sub>*(C)

*w* = 1/σ<sup>2</sup>(*F*)

(Δ/σ)<sub>max</sub> < 0.001

Δρ<sub>max</sub> = 2.66 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -2.65 e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Absolute configuration:

Flack (1983) parameter

= 0.028 (3)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

*U<sub>iso</sub>* for C atoms; *U<sub>eq</sub>* = (1/3)Σ<sub>*i*</sub>Σ<sub>*j*</sub>*U<sub>ij</sub>a<sub>i</sub><sup>\*</sup>a<sub>j</sub><sup>\*</sup>* for Pt, Cl, P and O.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub>/U<sub>iso</sub></i>
Pt(1)	-0.5030	-0.30321 (3)	-0.4997	0.02430 (10)
Cl(1)	-0.2659 (3)	-0.2998 (2)	-0.5315 (2)	0.0398 (10)
Cl(2)	-0.3966 (3)	-0.1063 (2)	-0.9121 (2)	0.0407 (10)
P(1)	-0.5064 (2)	-0.3645 (2)	-0.3797 (2)	0.0250 (8)
P(2)	-0.5731 (2)	-0.4420 (2)	-0.5191 (2)	0.0262 (7)
P(3)	-0.5401 (2)	-0.2637 (2)	-0.6316 (2)	0.0292 (8)
P(4)	-0.4367 (3)	-0.1725 (2)	-0.4449 (2)	0.0289 (7)
O(1)	-0.5760 (8)	-0.2343 (7)	-0.8771 (6)	0.054 (3)
O(2)	-0.5888 (8)	-0.0595 (7)	-0.0562 (6)	0.048 (3)
C(1)	-0.3851 (9)	-0.4190 (8)	-0.3307 (7)	0.029 (3)
C(2)	-0.3843 (9)	-0.4695 (8)	-0.2610 (7)	0.026 (3)
C(3)	-0.289 (1)	-0.5099 (9)	-0.2272 (8)	0.040 (3)
C(4)	-0.1979 (10)	-0.4995 (8)	-0.2568 (7)	0.032 (3)
C(5)	-0.198 (1)	-0.4496 (9)	-0.3217 (8)	0.038 (3)
C(6)	-0.2916 (9)	-0.4082 (8)	-0.3581 (7)	0.031 (3)
C(7)	-0.5634 (10)	-0.3094 (8)	-0.3084 (7)	0.032 (3)
C(8)	-0.671 (1)	-0.2782 (9)	-0.3316 (8)	0.042 (3)
C(9)	-0.719 (1)	-0.2335 (10)	-0.2808 (8)	0.045 (4)
C(10)	-0.664 (1)	-0.2219 (10)	-0.2047 (9)	0.046 (4)
C(11)	-0.558 (1)	-0.2504 (9)	-0.1807 (8)	0.041 (3)
C(12)	-0.509 (1)	-0.2917 (9)	-0.2309 (8)	0.040 (3)
C(13)	-0.6069 (9)	-0.4452 (8)	-0.4241 (7)	0.027 (3)

C(14)	-0.6923 (9)	-0.4649 (8)	-0.5945 (6)	0.024 (3)
C(15)	-0.790 (1)	-0.4266 (9)	-0.5922 (7)	0.034 (3)
C(16)	-0.883 (1)	-0.4411 (9)	-0.6563 (7)	0.036 (3)
C(17)	-0.8688 (10)	-0.4953 (8)	-0.7164 (7)	0.031 (3)
C(18)	-0.770 (1)	-0.5352 (9)	-0.7160 (8)	0.040 (3)
C(19)	-0.685 (1)	-0.5251 (9)	-0.6530 (8)	0.040 (3)
C(20)	-0.4790 (9)	-0.5216 (7)	-0.5174 (6)	0.025 (3)
C(21)	-0.495 (2)	-0.6047 (7)	-0.498 (1)	0.038 (3)
C(22)	-0.421 (1)	-0.6681 (9)	-0.4907 (8)	0.038 (3)
C(23)	-0.325 (1)	-0.6535 (10)	-0.5162 (8)	0.044 (4)
C(24)	-0.304 (1)	-0.5747 (10)	-0.5412 (8)	0.044 (3)
C(25)	-0.378 (1)	-0.5081 (9)	-0.5429 (7)	0.036 (3)
C(26)	-0.472 (1)	-0.175 (1)	-0.6691 (9)	0.052 (4)
C(27)	-0.677 (1)	-0.2371 (10)	-0.6683 (8)	0.043 (3)
C(28)	-0.516 (1)	-0.3515 (9)	-0.6948 (8)	0.041 (3)
C(29)	-0.333 (1)	-0.1859 (9)	-0.3564 (8)	0.036 (3)
C(30)	-0.539 (1)	-0.1093 (10)	-0.4148 (9)	0.049 (4)
C(31)	-0.370 (1)	-0.0971 (9)	-0.4993 (8)	0.044 (3)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Pt(1)—P(1)	2.317 (6)	P(2)—C(14)	1.81 (2)
Pt(1)—P(2)	2.352 (7)	P(2)—C(20)	1.73 (2)
Pt(1)—P(3)	2.331 (7)	P(3)—C(26)	1.84 (3)
Pt(1)—P(4)	2.345 (7)	P(3)—C(27)	1.77 (3)
P(1)—C(1)	1.80 (2)	P(3)—C(28)	1.83 (3)
P(1)—C(7)	1.79 (3)	P(4)—C(29)	1.80 (3)
P(1)—C(13)	1.84 (2)	P(4)—C(30)	1.80 (3)
P(2)—C(13)	1.80 (3)	P(4)—C(31)	1.84 (3)
P(1)—Pt(1)—P(2)	70.3 (2)	C(13)—P(2)—C(20)	105 (1)
P(1)—Pt(1)—P(3)	164.9 (2)	C(14)—P(2)—C(20)	109 (1)
P(1)—Pt(1)—P(4)	94.1 (2)	Pt(1)—P(3)—C(26)	123 (1)
P(2)—Pt(1)—P(3)	96.8 (2)	Pt(1)—P(3)—C(27)	112.9 (10)
P(2)—Pt(1)—P(4)	164.2 (2)	Pt(1)—P(3)—C(28)	111.7 (9)
P(3)—Pt(1)—P(4)	98.4 (3)	C(26)—P(3)—C(27)	101 (1)
Pt(1)—P(1)—C(1)	116.3 (9)	C(26)—P(3)—C(28)	101 (1)
Pt(1)—P(1)—C(7)	121.2 (9)	C(27)—P(3)—C(28)	103 (1)
Pt(1)—P(1)—C(13)	93.2 (8)	Pt(1)—P(4)—C(29)	112.0 (10)
C(1)—P(1)—C(7)	109 (1)	Pt(1)—P(4)—C(30)	112 (1)
C(1)—P(1)—C(13)	107 (1)	Pt(1)—P(4)—C(31)	121.2 (10)
C(7)—P(1)—C(13)	106 (1)	C(29)—P(4)—C(30)	104 (1)
Pt(1)—P(2)—C(13)	93.1 (8)	C(29)—P(4)—C(31)	100 (1)
Pt(1)—P(2)—C(14)	121.2 (8)	C(30)—P(4)—C(31)	103 (1)
Pt(1)—P(2)—C(20)	115.7 (8)	P(1)—C(13)—P(2)	94 (1)
C(13)—P(2)—C(14)	109 (1)		

Data collection used a Rigaku AFC-7S four-circle diffractometer equipped with an Oxford Systems low-temperature attachment. The temperature for data collection was 150 K. The Pt atom position was located by heavy-atom Patterson methods and the remaining non-H atoms were found from difference Fourier syntheses. As there were no identifiable faces, the data were corrected for absorption using *DIFABS* (Walker & Stuart, 1983). This correction was applied to the raw data with the model at isotropic convergence. In the final refinement calculations, anisotropic displacement parameters were adjusted only for Pt, Cl, P and O atoms. Fixed contributions for the scattering of methyl, methylene and phenyl H atoms [ $C-H = 0.96 \text{\AA}$ ] were added to the structure factors.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We thank the EPSRC for support and for a grant to purchase the diffractometer, and BP Chemicals and the EPSRC for supporting AFC.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1219). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.

## References

- Anderson, G. K. & Lumetta, G. J. (1987). *Inorg. Chem.* **26**, 1518–1524.
- Bernardinelli, G. & Flack, H. D. (1985). *Acta Cryst.* **A41**, 500.
- Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.
- Bhattacharyya, P., Sheppard, R. N., Slawin, A. M. Z., Williams, D. J. & Woollins, J. D. (1993). *J. Chem. Soc. Dalton Trans.* pp. 2393–2400.
- Braterman, P. S., Cross, R. J., Manojlović-Muir, L., Muir, K. W. & Young, G. B. (1975). *J. Organomet. Chem.* **84**, C40–42.
- Brüggeller, P., Nar, H. & Messerschmidt, A. (1992). *Acta Cryst.* **C48**, 817–821.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1992). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Steffan, W. L. & Palenik, G. J. (1976). *Inorg. Chem.* **15**, 2432–2439.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

*Acta Cryst.* (1996). **C52**, 801–803

## A Manganese Quinaldinate Complex: *trans*-[Diaquabis(2-quinolinecarboxylato)-manganese(II)]–Water–Ethanol (1/2/2)

HELMUT M. HAENDLER

*Department of Chemistry, University of New Hampshire, Durham, NH 03824-3598, USA*

(Received 22 March 1995; accepted 9 October 1995)

### Abstract

The title compound,  $[\text{Mn}(\text{C}_{10}\text{H}_6\text{NO}_2)_2(\text{H}_2\text{O})_2] \cdot 2\text{C}_2\text{H}_5\text{OH} \cdot 2\text{H}_2\text{O}$ , has a six-coordinate Mn atom at the center of symmetry, with two bidentate quinaldinate ligands and two water molecules in *trans* configurations. Two uncoordinated water molecules and two uncoordinated ethanol molecules stabilize the complex by participation in an extended hydrogen-bonding network.

### Comment

The structure analysis of the title compound, (I), is a continuation of the investigation of metal complexes of