Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

(Benzoylformaldehyde oximato-κ^2N,O)(benzoylformaldehyde oxime-κN)chloridoplatinum(II)

Nikolay V. Kukushkin,[a] Petru M. Ketrush[b] and Matti Haukka[c]*

[a]Department of Biochemistry, St Petersburg State University, St Petersburg, Russian Federation, [b]Department of Chemistry, Moldova State University, Chisinau, Moldova, and [c]Department of Chemistry, University of Joensuu, PO Box 111, FI-80101 Joensuu, Finland
Correspondence e-mail: matti.haukka@joensuu.fi

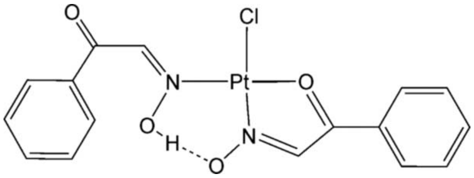
Received 29 March 2007; accepted 30 March 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.006 Å; *R* factor = 0.022; *wR* factor = 0.043; data-to-parameter ratio = 12.9.

In the title complex, $[Pt(C_8H_6NO_2)Cl(C_8H_7NO_2)]$, the Pt^{II} centre is coordinated by a monodentate and an *N*,*O*-chelating deprotonated benzoylformaldehyde oxime and a Cl atom. There is an intramolecular $N-O^- \cdots H-O$ hydrogen-bonding system between the oxime OH group and the oximate O atom.

Related literature

For related literature, see: Allen *et al.* (1987); Makarycheva-Mikhailova, Bokach *et al.* (2003); Makarycheva-Mikhailova, Haukka, Bokach *et al.* (2002); Raston *et al.* (1978).



Experimental

Crystal data

$[Pt(C_8H_6NO_2)Cl(C_8H_7NO_2)]$
M_r = 527.82
Monoclinic, $P2_1/c$
a = 7.1236 (2) Å
b = 18.0103 (6) Å
c = 12.5569 (4) Å
β = 97.163 (2)°
V = 1598.45 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.97 mm^{-1}
T = 100 (2) K
0.14 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*XPREP* in *SHELXTL*; Sheldrick, 2005)
T_{min} = 0.352, T_{max} = 0.636
16177 measured reflections
2855 independent reflections
2439 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.043
S = 1.06
2855 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.21 e Å^{-3}
$\Delta\rho_{min}$ = −1.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H03···O1	0.94 (7)	1.62 (7)	2.550 (4)	170 (6)

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Russian Fund for Basic Research (grant No. 06–03-90901). The authors thank Dr A. V. Makarycheva-Mikhailova for experimental assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2220).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Makarycheva-Mikhailova, A. V., Bokach, N. A., Haukka, M. & Kukushkin, V. Yu. (2003). *Inorg. Chim. Acta*, **356**, 382–386.
Makarycheva-Mikhailova, A. V., Haukka, M., Bokach, N. A., Garnovskii, D. A., Galanski, M., Keppler, B. K., Pombeiro, A. J. L. & Kukushkin, V. Yu. (2002). *New J. Chem. 26*, pp. 1085–1091.
Nonius (2004). *COLLECT*. Nonius BV, Delt, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Raston, C. L., Sharma, R. P., Skelton, B. W. & White, A. H. (1978). *Aust. J. Chem.* **31**, 745–755.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

doi:10.1107/S1600536807015747

supplementary materials

Acta Cryst. (2007). E**63**, m1286   [**doi:10.1107/S1600536807015747**]

(Benzoylformaldehyde oximato-κ^2*N*,*O*)(benzoylformaldehyde oxime-κ*N*)chloridoplatinum(II)

N. V. Kukushkin, P. M. Ketrush and **M. Haukka**

Comment

In the title complex, (I), a chloride anion is coordinated to the Pt^{II} centre, two sites are occupied by the deprotonated chelate, coordinated *via* the oximato nitrogen and the carbonyl oxygen, with the remaining site occupied by the monodentate oxo(phenyl)acetaldehyde oxime, which is coordinated *via* the oxime nitrogen, located at the *cis*-position to the oximato N atom. The bond lengths obtained for the monodentate ligand (Table 1) are similar to those reported for 1-oxo(phenyl)acetaldehyde oxime (N–O,1.380 (2) Å, N=C, 1.284 (2) Å, C–C,1.491 (3) Å, C=O, 1.224 (2) Å) (Raston *et al.*, 1978) These values are also in concord with the corresponding mean bond lengths values, obtained for N–O, N=C, C–C(unconjugated), C=O bonds: 1.394 (18), 1.281 (13), 1.478 (12), 1.210 (8) Å, respectively (Allen *et al.*, 1987). In contrast, the bond lengths found for the deprotonated N,*O*-chelated oxime (Table 1) differ from those observed in the monodentate ligand, the free molecule and from the standard bond lengths. We assume that the noticeable decrease in the N=C and C=O and concomitant increase in the N–O and C–C bond lengths in the N,*O*-chelate are a consequence of the availability of a coplanar conjugated system in the chelated ring, which stabilizes the oximato anion by delocalization of the negative charge. The structure is stabilized also by an intramolecular N—O^-···H–O hydrogen bond (Table 2).

The N=C–C=O torsion angles differ for the two ligands, being -0.1 (5)° for the chelated ligand and -100.1 (5)° for the monodentate species. The latter results in the phenyl plane being almost perpendicular to the coordination plane.

Experimental

The title compound is formed in small quantities as a by-product of the reaction between *trans*-$[PtCl_4(EtCN)_2]$ and the oxime leading mostly to *trans*-$[PtCl_4\{NH{=}C(Et)ON{=}C(H)C({=}O)Ph\}_2]$, followed by dehydration of the latter (Makarycheva-Mikhailova *et al.*, 2002, 2003).

Refinement

The OH hydrogen atom was located from the difference Fourier map and refined isotropically. Other H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.95 Å, and $U_{iso}(H) = 1.2\,U_{eq}$(parent atom). The highest peak is located 1.04Å from atom H16 and the deepest hole is located 0.86Å from atom Pt1.

Figures

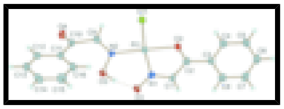


Fig. 1. A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The hydrogen is indicated by dashed line.

(Benzoylformaldehyde oximato-κ^2*N*,*O*)(benzoylformaldehyde oxime-κ*N*)chloridoplatinum(II)

Crystal data

$[Pt(C_8H_6NO_2)Cl(C_8H_7NO_2)]$

$M_r = 527.82$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.1236$ (2) Å

$b = 18.0103$ (6) Å

$c = 12.5569$ (4) Å

$\beta = 97.163$ (2)°

$V = 1598.45$ (9) Å^3

$Z = 4$

$F_{000} = 1000$

$D_x = 2.193$ Mg m^{-3}

Mo *K*α radiation
$\lambda = 0.71073$ Å

Cell parameters from 16177 reflections

$\theta = 2.3–25.2$°

$\mu = 8.97$ mm^{-1}

$T = 100$ (2) K

Needle, orange red

0.14 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: horizontally mounted graphite crystal

Detector resolution: 9 pixels mm^{-1}

$T = 100(2)$ K

φ scans and ω scans with κ offset

Absorption correction: multi-scan
(XPREP in SHELXTL; Sheldrick, 2005)

$T_{min} = 0.352$, $T_{max} = 0.636$

16177 measured reflections

2855 independent reflections

2439 reflections with $I > 2\sigma(I)$

$R_{int} = 0.051$

$\theta_{max} = 25.2$°

$\theta_{min} = 2.3$°

$h = -8 \rightarrow 8$

$k = -21 \rightarrow 21$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.022$

$wR(F^2) = 0.043$

$S = 1.06$

2855 reflections

221 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of
independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0141P)^2 + 1.3333P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 1.21$ e Å^{-3}

$\Delta\rho_{min} = -1.17$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1876 (5)	0.4606 (2)	−0.0671 (3)	0.0183 (9)
H1	0.1727	0.4803	−0.1379	0.022*
C2	0.2619 (5)	0.5028 (2)	0.0253 (3)	0.0181 (9)
C3	0.3210 (5)	0.5805 (2)	0.0197 (3)	0.0175 (9)
C4	0.3804 (6)	0.6181 (2)	0.1148 (4)	0.0239 (10)
H4	0.3826	0.5932	0.1816	0.029*
C5	0.4363 (6)	0.6916 (2)	0.1125 (4)	0.0281 (11)
H5	0.4765	0.7171	0.1775	0.034*
C6	0.4335 (6)	0.7279 (2)	0.0145 (4)	0.0278 (11)
H6	0.4711	0.7784	0.0129	0.033*
C7	0.3765 (6)	0.6910 (2)	−0.0801 (4)	0.0265 (10)
H7	0.3759	0.7161	−0.1467	0.032*
C8	0.3200 (5)	0.6175 (2)	−0.0786 (4)	0.0220 (10)
H8	0.2807	0.5922	−0.1439	0.026*
C9	0.0374 (5)	0.2189 (2)	0.1658 (3)	0.0210 (9)
H9	0.0588	0.2361	0.2378	0.025*
C10	−0.0491 (6)	0.1426 (2)	0.1463 (3)	0.0201 (9)
C11	0.0789 (5)	0.0790 (2)	0.1372 (3)	0.0197 (9)
C12	0.0063 (6)	0.0068 (2)	0.1389 (3)	0.0212 (10)
H12	−0.1236	−0.0008	0.1458	0.025*
C13	0.1232 (6)	−0.0531 (2)	0.1304 (3)	0.0247 (10)
H13	0.0743	−0.1021	0.1322	0.030*
C14	0.3121 (6)	−0.0421 (3)	0.1192 (4)	0.0296 (11)
H14	0.3919	−0.0837	0.1124	0.036*
C15	0.3855 (6)	0.0293 (3)	0.1179 (4)	0.0325 (11)
H15	0.5155	0.0364	0.1108	0.039*
C16	0.2703 (6)	0.0897 (3)	0.1270 (3)	0.0264 (10)
H16	0.3207	0.1385	0.1263	0.032*
N1	0.1419 (4)	0.39174 (18)	−0.0447 (3)	0.0193 (8)
N2	0.0835 (4)	0.26159 (18)	0.0929 (3)	0.0177 (7)
O1	0.0805 (4)	0.34699 (16)	−0.1200 (2)	0.0263 (7)
O2	0.2757 (4)	0.47114 (15)	0.1162 (2)	0.0186 (6)
O3	0.0550 (5)	0.23008 (18)	−0.0082 (3)	0.0316 (8)

H03	0.061 (8)	0.270 (4)	−0.056 (5)	0.08 (2)*
O4	−0.2187 (4)	0.13761 (15)	0.1456 (2)	0.0251 (7)
Cl1	0.26457 (14)	0.34896 (6)	0.28994 (8)	0.0236 (2)
Pt1	0.18476 (2)	0.365032 (8)	0.108499 (12)	0.01658 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.020 (2)	0.016 (2)	0.019 (2)	−0.0014 (17)	0.0037 (17)	0.0049 (18)
C2	0.009 (2)	0.018 (2)	0.028 (3)	0.0041 (16)	0.0037 (17)	0.0009 (19)
C3	0.015 (2)	0.016 (2)	0.021 (2)	0.0045 (16)	0.0032 (16)	0.0001 (18)
C4	0.022 (2)	0.023 (3)	0.027 (3)	−0.0002 (18)	0.0050 (18)	−0.0026 (19)
C5	0.029 (3)	0.023 (2)	0.032 (3)	−0.0059 (19)	0.005 (2)	−0.006 (2)
C6	0.023 (2)	0.018 (2)	0.043 (3)	0.0000 (18)	0.010 (2)	0.000 (2)
C7	0.021 (2)	0.025 (3)	0.034 (3)	0.0015 (19)	0.0084 (19)	0.010 (2)
C8	0.018 (2)	0.023 (2)	0.025 (3)	0.0024 (17)	0.0027 (18)	0.0009 (18)
C9	0.024 (2)	0.019 (2)	0.020 (2)	0.0011 (18)	0.0023 (18)	−0.0009 (19)
C10	0.029 (2)	0.016 (2)	0.015 (2)	−0.0036 (19)	0.0007 (17)	0.0043 (18)
C11	0.024 (2)	0.018 (2)	0.017 (2)	−0.0021 (18)	0.0010 (17)	0.0004 (18)
C12	0.022 (2)	0.023 (2)	0.018 (2)	−0.0027 (18)	0.0011 (17)	0.0003 (19)
C13	0.033 (3)	0.019 (2)	0.021 (3)	−0.0042 (19)	−0.0024 (19)	0.0018 (19)
C14	0.031 (3)	0.024 (3)	0.032 (3)	0.007 (2)	−0.003 (2)	−0.005 (2)
C15	0.020 (2)	0.038 (3)	0.039 (3)	0.001 (2)	0.002 (2)	−0.006 (2)
C16	0.026 (2)	0.024 (3)	0.028 (3)	−0.009 (2)	0.0015 (18)	−0.003 (2)
N1	0.0146 (17)	0.0199 (19)	0.024 (2)	−0.0008 (14)	0.0054 (14)	−0.0022 (16)
N2	0.0205 (18)	0.0172 (18)	0.0158 (19)	−0.0001 (15)	0.0036 (14)	−0.0017 (15)
O1	0.0374 (18)	0.0210 (17)	0.0201 (17)	−0.0086 (13)	0.0016 (13)	−0.0041 (13)
O2	0.0179 (14)	0.0163 (15)	0.0213 (17)	0.0022 (12)	0.0011 (11)	0.0006 (13)
O3	0.053 (2)	0.0237 (18)	0.0195 (18)	−0.0125 (15)	0.0093 (15)	−0.0039 (15)
O4	0.0198 (16)	0.0215 (16)	0.0342 (19)	−0.0010 (13)	0.0031 (13)	0.0031 (14)
Cl1	0.0298 (6)	0.0211 (6)	0.0192 (6)	−0.0015 (4)	−0.0001 (4)	0.0008 (4)
Pt1	0.01681 (9)	0.01454 (9)	0.01845 (10)	0.00001 (7)	0.00242 (6)	−0.00053 (8)

Geometric parameters (Å, °)

C1—N1	1.322 (5)	C10—C11	1.477 (6)
C1—C2	1.432 (6)	C11—C16	1.398 (6)
C1—H1	0.9500	C11—C12	1.401 (6)
C2—O2	1.270 (5)	C12—C13	1.375 (6)
C2—C3	1.465 (5)	C12—H12	0.9500
C3—C4	1.392 (6)	C13—C14	1.384 (6)
C3—C8	1.402 (6)	C13—H13	0.9500
C4—C5	1.383 (6)	C14—C15	1.389 (6)
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.391 (6)	C15—C16	1.377 (6)
C5—H5	0.9500	C15—H15	0.9500
C6—C7	1.378 (6)	C16—H16	0.9500
C6—H6	0.9500	N1—O1	1.278 (4)
C7—C8	1.384 (6)	N1—Pt1	1.970 (3)

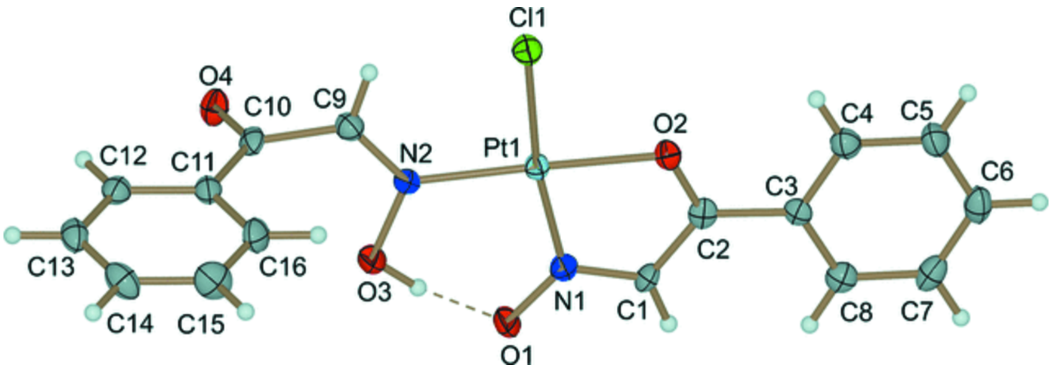
C7—H7	0.9500	N2—O3	1.383 (4)
C8—H8	0.9500	N2—Pt1	1.999 (3)
C9—N2	1.269 (5)	O2—Pt1	2.016 (3)
C9—C10	1.514 (6)	O3—H03	0.94 (7)
C9—H9	0.9500	Cl1—Pt1	2.2967 (10)
C10—O4	1.211 (5)		

N1—C1—C2	113.7 (4)	C16—C11—C10	121.3 (4)
N1—C1—H1	123.2	C12—C11—C10	119.1 (4)
C2—C1—H1	123.2	C13—C12—C11	119.9 (4)
O2—C2—C1	118.0 (3)	C13—C12—H12	120.0
O2—C2—C3	118.8 (4)	C11—C12—H12	120.0
C1—C2—C3	123.2 (4)	C12—C13—C14	120.1 (4)
C4—C3—C8	119.4 (4)	C12—C13—H13	119.9
C4—C3—C2	118.9 (4)	C14—C13—H13	119.9
C8—C3—C2	121.7 (4)	C13—C14—C15	120.3 (4)
C5—C4—C3	120.4 (4)	C13—C14—H14	119.8
C5—C4—H4	119.8	C15—C14—H14	119.8
C3—C4—H4	119.8	C16—C15—C14	120.1 (4)
C4—C5—C6	119.7 (4)	C16—C15—H15	119.9
C4—C5—H5	120.2	C14—C15—H15	119.9
C6—C5—H5	120.2	C15—C16—C11	119.8 (4)
C7—C6—C5	120.5 (4)	C15—C16—H16	120.1
C7—C6—H6	119.8	C11—C16—H16	120.1
C5—C6—H6	119.8	O1—N1—C1	120.3 (4)
C6—C7—C8	120.2 (4)	O1—N1—Pt1	124.5 (3)
C6—C7—H7	119.9	C1—N1—Pt1	115.1 (3)
C8—C7—H7	119.9	C9—N2—O3	113.1 (3)
C7—C8—C3	119.8 (4)	C9—N2—Pt1	128.1 (3)
C7—C8—H8	120.1	O3—N2—Pt1	118.8 (2)
C3—C8—H8	120.1	C2—O2—Pt1	113.2 (2)
N2—C9—C10	124.7 (4)	N2—O3—H03	105 (4)
N2—C9—H9	117.6	N1—Pt1—N2	97.01 (14)
C10—C9—H9	117.6	N1—Pt1—O2	79.94 (12)
O4—C10—C11	124.5 (4)	N2—Pt1—O2	176.47 (12)
O4—C10—C9	117.0 (4)	N1—Pt1—Cl1	171.42 (10)
C11—C10—C9	118.3 (3)	N2—Pt1—Cl1	91.22 (10)
C16—C11—C12	119.7 (4)	O2—Pt1—Cl1	91.91 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H03···O1	0.94 (7)	1.62 (7)	2.550 (4)	170 (6)

Fig. 1


Cl1
O4
C10
C9
C12
C11
N2
Pt1
O2
C4
C5
C6
C3
C2
C13
C16
O3
N1
C1
C14
C15
O1
C8
C7