Acta Cryst. (1990). C**46**, 137–138

Structure of (−)-(*R*)-[2-(Aminomethyl)pyrrolidine](1,1-cyclobutanedicarboxylato)-platinum(II) Monohydrate (DWA-2114R)

BY M. YONEI,† H. MURATA, M. ITOH, Y. WATANABE, K. OCHI, M. HONDA AND Y. NAWATA*

Research Laboratories, Chugai Pharmaceutical Co. Ltd, Takada, Toshima, Tokyo 171, *Japan*

(*Received* 13 *July* 1989; *accepted* 16 *August* 1989)

Abstract. $[Pt(C_6H_6O_4)(C_5H_{12}N_2)].H_2O$, $M_r = 455{\cdot}38$, monoclinic, $P2_1$, $a = 8{\cdot}7712\ (7)$, $b = 10{\cdot}720\ (1)$, $c = 7{\cdot}3221\ (6)$ Å, $\beta = 93{\cdot}03\ (1)°$, $V = 687{\cdot}5\ (2)$ Å^3, $Z = 2$, $D_x = 2{\cdot}200$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 103{\cdot}266$ cm^{-1}, $F(000) = 436$, $T = 298$ K, final $R = 0{\cdot}020$ for 2700 unique reflections $[F_o^2 > 2\sigma(F_o^2)]$. DWA-2114R is a square-planar Pt complex with the dicarboxylate chelate ring in a boat conformation and with the aminomethylpyrrolidine chelate ring in an envelope conformation. The cyclobutane ring adopts a puckered conformation. The absolute configuration was determined by the Bijvoet method.

† Deceased.

* To whom correspondence should be addressed.

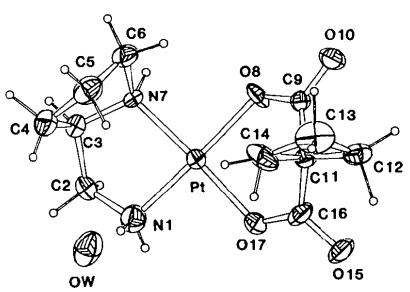

Fig. 1. A perspective view of the molecule with the numbering scheme.

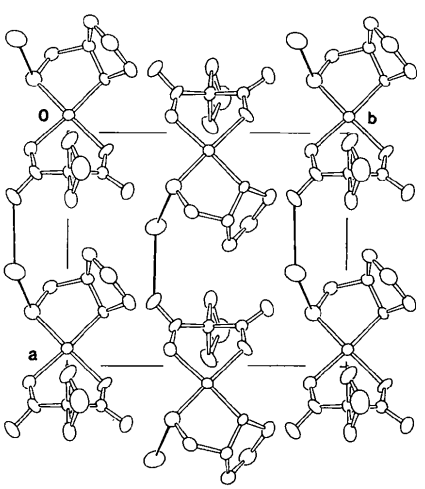

Fig. 2. Packing diagram for the title compound. Hydrogen bonds are shown as narrow lines.

Experimental. Colorless prisms of title compound were grown from an aqueous solution. $[\alpha]_D^{20°C} = -41{\cdot}7°$ (water). Crystal size 0·20 × 0·13 × 0·10 mm, Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, θ–2θ scan with scan speed 1·65–4·12° min^{-1} in θ, scan width $(0{\cdot}50 + 0{\cdot}15\tan\theta)°$. Range of indices, $-14 \le h \le 14$, $0 \le k \le 17$, $0 \le l \le 11$ ($\theta < 35°$). Lattice constants determined based on 25 2θ values ($11 < \theta < 18°$). Variation of standard <2·5%; 3340 reflections measured; 2700 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. Systematic absences $0k0$, k odd. Empirical corrections for absorption (North, Phillips & Mathews, 1968); min., max. transmission coefficients 0·937, 1·000. Structure solved by the heavy-atom method. Refined by full-matrix least squares. The locations of 18 H atoms were calculated. Non-H atoms refined with anisotropic thermal parameters, but H atoms with fixed isotropic thermal parameters ($B = 5{\cdot}0$ Å^2). $\sum w(|F_o|$

Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	B_{eq}(Å^2)
Pt	0·92524 (2)	1·000	0·58856 (2)	1·989 (2)
N(1)	0·7701 (6)	0·8759 (6)	0·4950 (8)	3·3 (1)
C(2)	0·6641 (6)	0·9400 (5)	0·3607 (8)	2·83 (9)
C(3)	0·6262 (6)	1·0699 (5)	0·4250 (8)	2·56 (9)
C(4)	0·5219 (7)	1·0766 (6)	0·587 (1)	3·5 (1)
C(5)	0·6118 (8)	1·1450 (7)	0·741 (1)	3·7 (1)
C(6)	0·7268 (7)	1·2185 (6)	0·6410 (9)	3·2 (1)
N(7)	0·7694 (5)	1·1275 (4)	0·4982 (6)	1·98 (6)
O(8)	1·0797 (4)	1·1334 (4)	0·6659 (6)	2·75 (7)
C(9)	1·1725 (5)	1·1217 (5)	0·8125 (6)	1·89 (7)
O(10)	1·2508 (5)	1·2118 (4)	0·8598 (6)	3·24 (8)
C(11)	1·1709 (4)	1·0029 (8)	0·9159 (5)	2·11 (6)
C(12)	1·2849 (6)	1·0094 (6)	1·0857 (6)	2·87 (9)
C(13)	1·153 (1)	1·0446 (8)	1·204 (1)	4·8 (2)
C(14)	1·0350 (6)	1·004 (1)	1·0514 (7)	3·73 (9)
O(15)	1·2779 (5)	0·8066 (4)	0·8300 (7)	3·74 (9)
C(16)	1·1740 (6)	0·8844 (5)	0·7924 (8)	2·80 (9)
O(17)	1·0723 (4)	0·8657 (4)	0·6677 (6)	2·89 (7)
OW	0·5986 (6)	0·8125 (6)	0·8261 (7)	4·7 (1)

0108-2701/90/010137-02$03.00

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Pt—N(1)	1·998 (5)	C(3)—N(7)	1·475 (7)	C(11)—C(12)	1·555 (6)
Pt—N(7)	2·019 (4)	C(4)—C(5)	1·532 (10)	C(11)—C(14)	1·591 (6)
Pt—O(8)	2·030 (4)	C(5)—C(6)	1·501 (10)	C(11)—C(16)	1·560 (10)
Pt—O(17)	1·998 (4)	C(6)—N(7)	1·492 (8)	C(12)—C(13)	1·529 (10)
N(1)—C(2)	1·486 (8)	O(8)—C(9)	1·318 (6)	C(13)—C(14)	1·546 (10)
C(2)—C(3)	1·512 (8)	C(9)—O(10)	1·224 (6)	O(15)—C(16)	1·255 (7)
C(3)—C(4)	1·535 (9)	C(9)—C(11)	1·482 (9)	C(16)—O(17)	1·258 (7)

N(1)—Pt—N(7)	84·4 (2)	O(8)—C(9)—C(11)	118·4 (4)
N(1)—Pt—O(8)	175·7 (2)	O(10)—C(9)—C(11)	123·6 (4)
N(1)—Pt—O(17)	92·1 (2)	C(9)—C(11)—C(12)	110·3 (5)
N(7)—Pt—O(8)	92·6 (2)	C(9)—C(11)—C(14)	110·0 (6)
N(7)—Pt—O(17)	176·3 (2)	C(9)—C(11)—C(16)	113·7 (4)
O(8)—Pt—O(17)	90·9 (2)	C(12)—C(11)—C(14)	88·5 (3)
N(1)—C(2)—C(3)	111·1 (5)	C(12)—C(11)—C(16)	118·0 (5)
C(2)—C(3)—C(4)	115·7 (5)	C(14)—C(11)—C(16)	113·7 (6)
C(2)—C(3)—N(7)	107·6 (4)	C(11)—C(12)—C(13)	89·4 (4)
C(4)—C(3)—N(7)	103·4 (5)	C(12)—C(13)—C(14)	91·1 (5)
C(3)—C(4)—C(5)	106·7 (5)	C(11)—C(14)—C(13)	87·5 (5)
C(4)—C(5)—C(6)	102·9 (6)	C(11)—C(16)—O(15)	116·7 (5)
C(5)—C(6)—N(7)	101·5 (5)	C(11)—C(16)—O(17)	121·1 (5)
C(3)—N(7)—C(6)	106·7 (4)	O(15)—C(16)—O(17)	122·1 (5)
O(8)—C(9)—O(10)	117·9 (5)		

$-|F_c|)^2$ minimized; $w = 1·0$ for $|F_o| < 119·26$, $w = (119·26/F_o)^2$ for $|F_o| \geq 119·26$. Final $R = 0·020$, $wR = 0·021$, $S = 1·88$ for 245 variables, secondary-extinction factor (g) $9·8\,(1) \times 10^{-7}$ $[|F_o| = |F_c|/(1 + gI_c)]$; $\Delta/\sigma < 0·48$ for non-H atoms, largest peak in final ΔF map $+1·3$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1984), *ORTEP*II (Johnson, 1976). The structure of the title compound is shown in Fig. 1, and the crystal packing in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, bond distances and angles are listed in Table 2.*

Related literature. The title compound is a low-toxicity antitumor Pt complex (Mitsui, Akamatsu, Koizumi, Tsuchiya, Tomita & Matsuno, 1987; Morikawa, Honda, Matsumoto, Endoh, Akamatsu, Mitsui & Koizumi, 1988). For the preparation of the compound see Morikawa, Honda & Endoh (1987).

* Lists of anisotropic thermal parameters, H-atom coordinates, torsion angles, least-squares planes, r.m.s. amplitudes of thermal vibration and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52209 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

FRENZ, B. A. (1984). *Enraf–Nonius Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MITSUI, H., AKAMATSU, K., KOIZUMI, M., TSUCHIYA, M., TOMITA, E. & MATSUNO, T. (1987). Presented at the 15th Int. Congr. Chemotherapy, Istanbul.

MORIKAWA, K., HONDA, M. & ENDOH, K. (1987). *Kokai Tokkyo Koho*, 61-129289 (Japanese patent pending).

MORIKAWA, K., HONDA, M., MATSUMOTO, T., ENDOH, K., AKAMATSU, K., MITSUI, H. & KOIZUMI, M. (1988). *Yakugaku Zasshi*, **108**, 797–800.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Acta Cryst. (1990). C**46**, 138–140

Bromodicarbonyl(η^3-1-phenylallyl)bis(pyrazole)molybdenum(II)

BY F. ALBERT COTTON* AND RUDY L. LUCK

Laboratory for Molecular Structure and Bonding, Texas A&M University, College Station, TX 77843, *USA*

(*Received* 27 *June* 1989; *accepted* 31 *July* 1989)

Abstract. $[MoBr(C_9H_9)(C_3H_4N_2)_2(CO)_2]$, $M_r = 485·19$, triclinic, $P\bar{1}$, $a = 8·149\,(1)$, $b = 9·418\,(1)$, $c = 12·917\,(2)$ Å, $\alpha = 79·91\,(1)$, $\beta = 80·87\,(1)$, $\gamma = 80·23\,(1)°$, $V = 953·4\,(3)$ Å^3, $Z = 2$, $D_x = 1·690$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 27·640$ cm^{-1}, $F(000) = 476$, $T = 292$ K, $R = 0·0689$ for 1776 unique observed reflections. The complex can be considered as octahedral if one assumes that the 1-phenylallyl ligand occupies only one coordination site. A pyrazolyl ligand is *trans* to the 1-phenylallyl ligand and the remaining ligands (one pyrazole, one bromide and two carbon monoxide ligands) can then be described as occupying equatorial positions with the two carbon monoxide ligands *cis* to each other. Bonds to Mo are Mo—Br = 2·756 (2), Mo—N(pyrazole *trans* to 1-phenylallyl) = 2·227 (11), Mo—N(pyrazole *cis* to 1-phenylallyl) = 2·266 (12), Mo—C(CO *trans* to Br) = 1·938 (15), Mo—C(CO *cis*

* To whom correspondence should be addressed.

0108-2701/90/010138-03$03.00