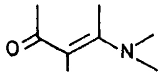
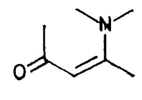
Palladium(II) Complexation of Ambidentate *cis*-*s*-*cis*-Enaminones: Synthesis of Some Bis(4-anilinopent-3-en-2-onato)- and Bis(3-amino-1-phenylbut-2-en-1-onato)-complexes of Palladium(II) and *X*-Ray Crystal Structure of Bis[4-(2-bromoanilino)pent-3-en-2-onato]palladium(II)

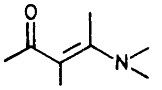
By **Hideo Iida, Yoshifumi Yuasa,** and **Chihiro Kibayashi,*** Tokyo College of Pharmacy, Horinouchi, Hacioji, Tokyo 192–03, Japan
Yoichi Iitaka,* Faculty of Pharmaceutical Science, Tokyo University, Hongo, Bunkyo-ku, Tokyo 113, Japan

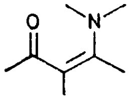
Condensation of acetylacetone with anilines gives *N*-aryl derivatives of 4-aminopent-3-en-2-one. Similar treatment of benzoylacetone with various primary amines yields *N*-substituted 3-amino-1-phenylbut-2-en-1-ones. Both series of products with the *cis*-*s*-*cis*-enaminone structure behave as bidentate ligands for Pd^{II} to yield stable yellow complexes, the structures of which have been determined as bis(4-anilinopent-3-en-2-onato)- and bis(3-amino-1-phenylbut-2-en-1-onato)-complexes of palladium(II), respectively. To establish the structure of the complex in the solid state, *X*-ray crystal structure analyses have been carried out on bis[4-(2-bromoanilino)pent-3-en-2-onato]palladium(II). The crystals of the complex solvated by benzene are triclinic and belong to space group $P\bar{1}$, with cell dimensions a = 8.034(5), b = 11.613(5), c = 7.637(10) Å, α = 98.67(5), β = 88.44(5), γ = 100.22(10)°, and Z = 1. The crystal structure has been solved using 2 951 reflections and refined to a final R value of 0.092. The Pd atom takes a square-planar co-ordination and the ligand molecules with the *cis*-*s*-*cis*-enaminone system form six-membered chelate rings with the metal.

In the enaminone (vinylogous amide) system the atoms comprising the π system lie in the same plane allowing maximum resonance stability, and thus four geometric isomers, *trans*-*s*-*trans*, *cis*-*s*-*trans*, *trans*-*s*-*cis*, and *cis*-*s*-*cis*, are possible (see below). These isomers can be


trans - *s* - *trans*


cis - *s* - *trans*


trans - *s* - *cis*


cis - *s* - *cis*

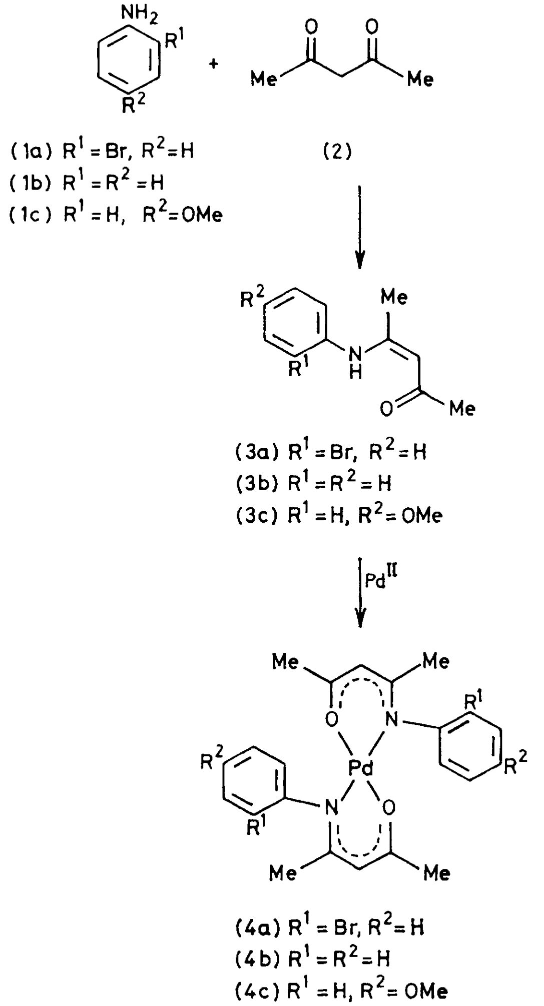
distinguished by u.v. spectroscopic data,[1] with difficulty of the distinction of the *cis*-*s*-*cis* form from the *cis*-*s*-*trans* form.[2] In the course of studies on the reaction of enaminones with palladium compounds, we discovered that enaminone molecules derived from β-diketones and primary amines with the *cis*-*s*-*cis* structure behaved as bidentate ligands for Pd^{II} to give stable crystalline complexes. Although there have been many reports on complexes prepared by similar ligands with other metals,[3] a search of the literature reveals no example of complex formation with palladium. Here we describe new preparations of some bis(4-anilinopent-3-en-2-onato)- and bis(3-amino-1-phenylbut-2-en-1-onato)-palladium(II) complexes, together with *X*-ray structural data on one of them.

RESULTS AND DISCUSSION

It is known that the enaminone system, $>N-\overset{|}{C}_{\beta}=\overset{|}{C}_{\alpha}-\overset{|}{C}=O$, possesses three possible reaction sites, O, C_α, and N, toward electrophiles and this has extensively been demonstrated by ordinary ionic reactions.[4] However, there has been no report of the reaction of the enaminone compounds involving organopalladium complexes except for our recent work on the intramolecular Heck arylation reaction [5] and on other ring closing reactions [6] carried out on cyclic enaminones with the *trans*-*s*-*trans* configuration. Following this, we were interested in attempting to apply the Heck reaction to acyclic enaminones with the *cis*-*s*-*cis* configuration. Thus the bromoenaminone (3a), prepared by condensation of *o*-bromoaniline (1a) with acetylacetone (2), was treated under the conditions of the Heck reaction: a mixture of (3a), palladium(II) acetate, triphenylphosphine, and $Na[HCO_3]$ in dimethylformamide (dmf) was heated at 120—130 °C for 24 h. After work-up, column chromatography on silica gel and recrystallisation from benzene afforded a stable crystalline complex with a characteristic yellow colour in 48% yield, instead of the arylation product. The isolated complex was shown to be bis[4-(2-bromoanilino)pent-3-en-2-onato]palladium(II) (4a) by spectroscopic and elemental analyses. The assigned structure (4a) was confirmed by *X*-ray crystal structure determination (see below).

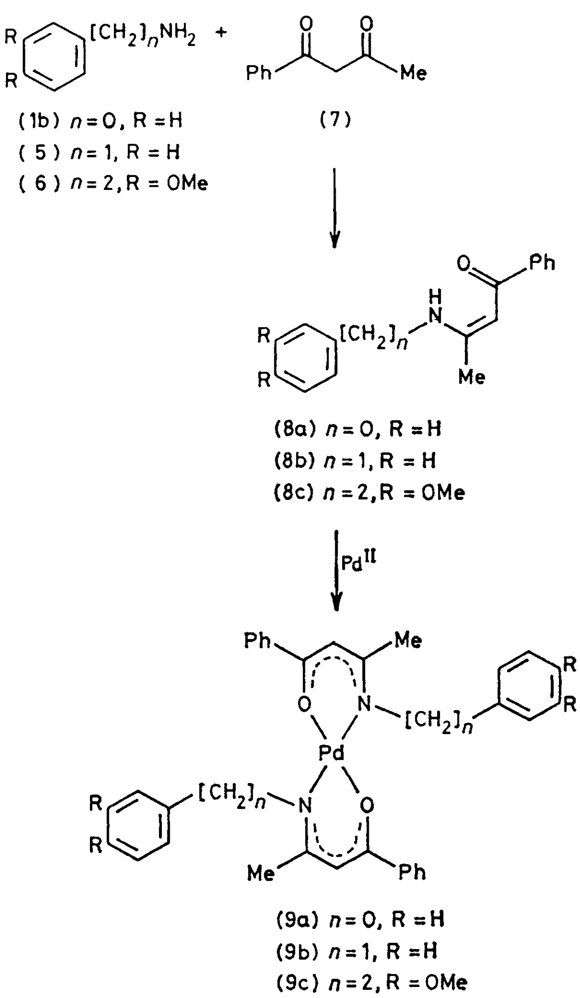
For a generalised procedure for the complexation of *N*-arylenaminones with the *cis*-*s*-*cis* structure, 2 mol equivalents of (3b) were treated with palladium(II) acetate in 1,2-dichloroethane at 50—60 °C for 7 h. The crude product was purified by column chromatography and recrystallisation yielded the palladium complex (4b) as yellow needles in 67% yield. In a similar way the complexation of (3c), available from *p*-anisidine (1c) and acetylacetone (2), afforded (4c) in 72% yield.

In another method of preparation of the enaminone–palladium complexes, the primary amines, aniline (1b), benzylamine (5), and 2-(3,4-dimethoxyphenyl)ethylamine (6), were condensed with benzoylacetone (7) to give the corresponding *cis*-*s*-*cis*-enaminones (8a), (8b), and (8c), respectively. When these enaminones were reacted with palladium(II) acetate in the molar ratio of 2 : 1 in refluxed chloroform for 12—20 h, the corresponding complexes (9a)—(9c) were obtained in 71—80% yields.



In conclusion, the complex formation of *cis-s-cis*-enaminones due to their ambidentate character can clearly be recognised both by isolation of stable crystalline products and their characteristic yellow colouration, and, as well as complexation with other metals such as Cu^{II}, Ni^{II}, and Co^{II}, may serve as a reliable method for distinction of *cis-s-cis*-enaminones from other isomers.*

Crystal Structure of $[Pd\{N(C_6H_4Br\text{-}o)C(Me)CHC(Me)O\}_2]\cdot C_6H_6$.—The molecular structure of the complex (4a) is shown in the Figure and bond distances and angles are given in Table 2. The Pd atom takes a square-planar co-ordination and the ligand molecules form six-membered chelate rings. The rings are planar within ±0.036 Å (Table 3) and the substituent benzene ring makes an angle of 88.7° with the chelate ring to avoid the repulsions from C(5) and O'. The bond lengths along the backbone of the ligand molecule indicated that the bonds C(2)=O and C(3)=C(4) are considerably elongated (by about 0.08 Å), while C(2)-C(3) and C(4)-N are shortened (by about 0.15 Å). This may well be accounted for by a conjugation of the double bonds along the backbone atoms. The widening of the bond angles O-C(2)-C(3), C(2)-C(3)-C(4), and C(3)-C(4)-N by about 5° over those expected for the usual conjugated system may be the consequence of the formation of a six-membered chelate ring around the Pd atom.



EXPERIMENTAL

Infrared spectra were recorded on a Hitachi 215 grating spectrophotometer for solutions in chloroform unless otherwise stated. Hydrogen-1 n.m.r. spectra in $CDCl_3$ were obtained on a JEOL JNM-PS-100 spectrometer with $SiMe_4$

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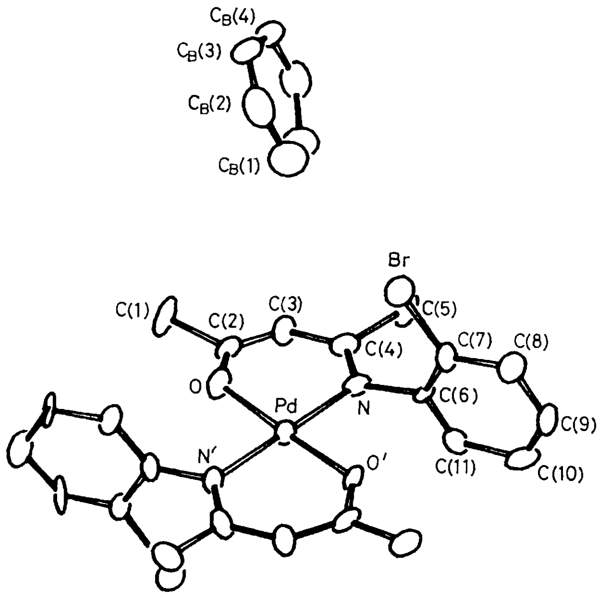
* A referee has suggested that in the case of the *cis-s-trans* structure the stable Pd complex could also be formed through its isomerisation to the *cis-s-cis* structure during complexation. In practice, however, it seems not to be necessary to take account of this, because usually acyclic enaminones are not able to exist in the *cis-s-trans* form owing to its rapid isomerisation and, thus, the *cis-s-trans* configuration has been found only in fixed heterocyclic systems such as 2,3-dihydro-4-pyridones (see, for example, refs. 1 and 2) where isomerisation to other configurations is prevented.

as internal reference. Mass spectra were taken with a Hitachi RMU-7L double-focusing spectrometer at 70 eV. Melting points were determined with a Yanagimoto micro apparatus and are uncorrected.

4-(2-*Bromoanilino*)*pent*-3-*en*-2-*one* (3a).—A mixture of *o*-bromoaniline (1a) (3.0 g, 0.018 mol) and acetylacetone (2) (3.0 g, 0.018 mol) was heated on an oil bath at 110 °C for 3 h. After cooling, the reaction mixture was dissolved in chloroform and this solution was washed with 5% HCl and dried (Mg[SO$_4$]). The solvent was removed by evaporation and the residue was chromatographed on a silica-gel column with benzene to give the crystalline *product* (3a) (1.6 g, 35%), m.p. 72—74 °C (Found: C, 52.35; H, 4.85; N, 5.5. $C_{11}H_{12}BrNO$ requires C, 52.0; H, 4.7; N, 5.5%); $\nu_{max.}$ (neat) at 3 450s (NH), 3 350s (NH), 3 175m (NH), 1 610 (sh), 1 605vs (C=O), and 1 585m cm^{-1} (C=C); ^{1}H n.m.r., τ 8.13 (s, 3 H, NCMe), 7.90 (s, 3 H, COMe), 4.78 (s, 1 H, vinylic), 3.00—2.75 (m, 3 H, aromatic), 2.50—2.33 (m, 1 H, aromatic), and −2.38 (s br, 1 H, NH); *m/e* 255 [M^+ ($C_{11}H_{12}{}^{79}BrNO$) + 2, 17%], 253 (M^+, 16), 240 (24), 238 (31), 174 (M^+ − Br, 100), 159 (54), and 131 (30).



Crystal structure of $[\mathrm{Pd}\{\mathrm{N}(\mathrm{C_6H_4Br}\text{-}o)\mathrm{C(Me)CHC(Me)O}\}_2]\cdot\mathrm{C_6H_6}$ showing the atom numbering scheme

4-*Anilinopent*-3-*en*-2-*one* (3b).—A mixture of aniline (1b) (3.0 g, 0.032 mol) and acetylacetone (2) (3.2 g, 0.032 mol) in benzene (50 cm^3) was heated under reflux using a Dean–Stark trap for azeotropic removal of water for 3 h. The benzene solution was washed with 5% HCl and dried (Mg[SO$_4$]). The crude product obtained by evaporation of the solvent was chromatographed on a silica-gel column with benzene to give a pale yellow *oil* (2.1 g, 38%); $\nu_{max.}$ (neat) at 3 425m (NH), 3 325m (NH), 3 200m (NH), 1 610s (sh), 1 600vs (C=O), and 1 560s cm^{-1} (C=C).

4-(4-*Methoxyanilino*)*pent*-3-*en*-2-*one* (3c).—A mixture of *p*-anisidine (1c) (2.1 g, 0.017 mol) and acetylacetone (2) (1.7 g, 0.017 mol) in benzene (40 cm^3) was treated in the same manner as that described above for (3b). After work-up, chromatography on a silica-gel column with benzene afforded a pale yellow *oil* (1.6 g, 46%); $\nu_{max.}$ (neat) at 3 400m, vbr (NH), 3 225—3 100m, vbr (NH), 1 610s (sh), 1 600vs (C=O), and 1 560s cm^{-1} (C=C).

Reaction of 4-(2-*Bromoanilino*)*pent*-3-*en*-2-*one* (3a) *under Heck Reaction Conditions: Formation of Bis*[4-(2-*bromoanilino*)*pent*-3-*en*-2-*onato*]*palladium*(II) (4a).—A mixture of the bromoenaminone (3a) (1.4 g) and Pd[O$_2$CMe]$_2$ (50 mg), triphenylphosphine (115 mg), and Na[HCO$_3$] (924 mg) in dmf (50 cm^3) was heated at 120—130 °C with stirring for 24 h. The reaction mixture was filtered through Celite and evaporated *in vacuo* to dryness. The residue was extracted with benzene and the benzene solution was washed with water and dried (Mg[SO$_4$]). After removal of the solvent by evaporation the residue was chromatographed on a silica-gel column with benzene. The first elution afforded the complex (4a) as yellow *needles* [65 mg, 48% (based on Pd)], m.p. 225—227.5 °C (from benzene) (Found: C, 43.2; H, 3.6; N, 4.7. $C_{22}H_{22}Br_2N_2O_2Pd$ requires C, 43.15; H, 3.60; N, 4.55%); $\nu_{max.}$ at 1 565s cm^{-1}; ^{1}H n.m.r., τ 8.61 (s, 6 H, NCMe), 8.37 (s, 6 H, COMe), 5.14 (s, 2 H, vinylic), and 3.07—2.44 (m, 8 H, aromatic); *m/e* 618 (7%), 617 (7), 616 (28), 615 (13), 614 (51), 613 (29), 612 [M^+ (as $C_{22}H_{22}{}^{79}Br_2N_2O_2{}^{106}Pd$), 51], 611 (34), 610 (30), 609 (15), 358 (29), 279 (31), 196 (24), 173 (48), and 130 (100).

Further elution yielded the unchanged starting material (3a) (460 mg).

Bis(4-*anilinopent*-3-*en*-2-*onato*)*palladium*(II) (4b).—A mixture of the enaminone (3b) (180 mg, 1.027 mmol) and Pd[O$_2$CMe]$_2$ (115 mg, 0.512 mmol) in 1,2-dichloroethane (15 cm^3) was stirred at 50—60 °C for 7 h. The reaction mixture was washed with water and the solvent was removed by evaporation *in vacuo*. The residue was purified by chromatography on silica gel with benzene and recrystallisation from benzene–hexane to give the complex (4b) as yellow *needles* which could be sublimed (155 mg, 67%), m.p. 243—246 °C (Found: C, 58.2; H, 5.3; N, 6.15. $C_{22}H_{24}N_2O_2Pd$ requires C, 58.1; H, 5.3; N, 6.15%); $\nu_{max.}$ at 1 565s cm^{-1}; ^{1}H n.m.r., τ 8.34 (s, 6 H, NCMe), 8.63 (s, 6 H, COMe), 5.20 (s, 2 H, vinylic), and 3.13—2.69 (m, 10 H, aromatic).

Bis[4-(4-*methoxyanilino*)*pent*-3-*en*-2-*onato*]*palladium*(II) (4c).—A mixture of the enaminone (3c) (170 mg, 0.828 mmol) and Pd[O$_2$CMe]$_2$ (93 mg, 0.414 mmol) was treated in the same manner as that described above for (4b). Purification by chromatography (silica gel, benzene) and recrystallisation from benzene–hexane gave (4c) as yellow *needles* which could be sublimed (154 mg, 72%), m.p. 254—256 °C (Found: C, 55.9; H, 5.5; N, 5.25. $C_{24}H_{28}N_2O_4Pd$ requires C, 56.0; H, 5.5; N, 5.45%); $\nu_{max.}$ at 1 590m and 1 560s cm^{-1}; ^{1}H n.m.r., τ 8.56 (s, 6 H, NCMe), 8.34 (COMe), 6.22 (s, 6 H, OMe), 5.20 (s, 2 H, vinylic), and 3.22 and 3.16 (AB, quartet, each 4 H, *J* 10 Hz, aromatic *o*-H and *m*-H to OMe, respectively).

3-*Anilino*-1-*phenylbut*-2-*en*-1-*one* (8a).—A mixture of aniline (1b) (580 mg, 6.2 mmol) and benzoylacetone (7) (1.00 g, 6.2 mmol) in benzene was treated in a manner similar to that described for the preparation of (3b). The crude product obtained by evaporation of the solvent was recrystallised from benzene–hexane to give (8a) as pale yellow *scales* (1.10 g, 75%), m.p. 110.5—111.5 °C (Found: C, 80.7; H, 6.35; N, 5.8. $C_{16}H_{15}NO$ requires C, 81.0; H, 6.35; N, 5.9%); $\nu_{max.}$ at 3 600—3 150m, vbr (NH), 1 600 (sh), 1 590s, and 1 570s cm^{-1}; ^{1}H n.m.r., τ 7.86 (s, 3 H, Me) and 4.14 (s, 1 H, vinylic).

3-*Benzylamino*-1-*phenylbut*-2-*en*-1-*one* (8b).—A mixture of benzylamine (5) (660 mg, 6.2 mmol) and benzoylacetone (7) (1.00 g, 6.2 mmol) in benzene was worked up in a similar manner to that described for (3b). The crude product obtained by evaporation of the solvent was recrystallised from benzene–hexane to give (8b) as colourless *needles* (1.35 g, 87%), m.p. 58—60 °C (Found: C, 80.95; H, 6.55; N, 5.6. $C_{17}H_{17}NO$ requires C, 81.25; H, 6.8; N, 5.55%); $\nu_{max.}$ at 3 630—3 150m, vbr (NH), 1 605 (sh), 1 590s, and 1 580 cm^{-1}; ^{1}H n.m.r., τ 7.92 (s, 3 H, Me), 5.44 (d, 2 H, *J* 5 Hz, C*H*$_2$Ph), 4.29 (s, 1 H, vinylic), and −1.90 (s br, 1 H, NH).

TABLE 1

Fractional atomic co-ordinates (× 10^5 for Br; × 10^4 for O, N, and C) for (4a), with estimated standard deviations in parentheses

Atom	*x*	*y*	*z*
Pd	0	0	0
Br	6 803(16)	33 574(13)	−7 028(20)
O	1 630(9)	10(7)	1 889(9)
N	−974(10)	1 352(7)	1 413(10)
C(1)	3 051(16)	410(13)	4 640(17)
C(2)	1 685(12)	612(9)	3 434(13)
C(3)	725(15)	1 461(10)	3 996(15)
C(4)	−507(13)	1 839(9)	3 054(13)
C(5)	−1 305(19)	2 878(13)	3 935(18)
C(6)	−2 171(10)	1 866(8)	516(12)
C(7)	−1 655(13)	2 740(9)	−469(13)
C(8)	−2 815(15)	3 223(11)	−1 399(14)
C(9)	−4 527(15)	2 795(12)	−1 266(17)
C(10)	−5 077(14)	1 877(12)	−268(15)
C(11)	−3 882(13)	1 432(10)	660(14)
$C_B(1)$	4 013(20)	4 165(13)	3 770(18)
$C_B(2)$	5 658(22)	4 694(14)	3 297(20)
$C_B(3)$	6 584(21)	5 495(16)	4 579(24)

3-[2-(3,4-*Dimethoxyphenyl)ethylamino*]-1-*phenylbut*-2-*en*-1-*one* (8c).—A mixture of 2-(3,4-dimethoxyphenyl)ethylamine (6) (1.12 g, 6.2 mmol) and benzoylacetone (7) (1.00 g, 6.2 mmol) in benzene was treated in a manner similar to that described for (3c). The crystals obtained by evaporation of the solvent was recrystallised from benzene–hexane to give (8c) as colourless fine *needles* (1.89 g, 94%), m.p. 81.5—83 °C (Found: C, 73.85; H, 7.0; N, 4.3. $C_{20}H_{23}NO_3$ requires C, 73.85; H, 7.1; N, 4.3%); $\nu_{max.}$ at 3 630—3 120m, vbr (NH), 1 605 (sh), 1 595vs (C=O), and 1 580s cm^{-1} (C=C). ^{1}H N.m.r., τ 8.13 (s, 3 H, CMe), 7.15 (t, 2 H, *J* 7, C*H*$_2$Ph), 6.47 (quartet, 2 H, *J* 7 Hz, NCH$_2$), 6.18 (s, br, 6 H, OMe), and 4.41 (s, 1 H, vinylic).

Bis(3-*anilino*-1-*phenylbut*-2-*en*-1-*onato*)*palladium*(II) (9a).—A mixture of the enaminone (8a) (272 mg, 1.146 mmol) and Pd[O_2CMe]$_2$ (128 mg, 0.570 mmol) in chloroform (25 cm^3) was refluxed with stirring for 20 h. The reaction mixture was evaporated to dryness and the residue was recrystallised from benzene to give (9a) as yellow *scales* (257 mg, 78%), m.p. >280 °C (Found: C, 66.3; H, 4.85; N, 4.7. $C_{32}H_{28}N_2O_2Pd$ requires C, 66.4; H, 4.85; N, 4.85%); $\nu_{max.}$ at 1 580m and 1 560m cm^{-1}.

Bis(3-*benzylamino*-1-*phenylbut*-2-*en*-1-*onato*)*palladium*(II) (9b).—A mixture of the enaminone (8b) (500 mg, 1.989 mmol) and Pd[O_2CMe]$_2$ (223 mg, 0.993 mmol) in chloroform (70 cm^3) was refluxed for 15 h. Usual work-up, as described above for (9a), and recrystallisation from benzene afforded (9b) as yellow fine *crystals* (428 mg, 71%), m.p. 259—261 °C (Found: C, 67.2; H, 5.3; N, 4.65. $C_{34}H_{32}N_2O_2Pd$ requires C, 67.25; H, 5.3; N, 4.6%); $\nu_{max.}$ at 1 590m and 1 570w cm^{-1}.

Bis{3-[2-(3,4-*dimethoxyphenyl)ethylamino*]-1-*phenylbut*-2-*en*-1-*onato*}*palladium*(II) (9c).—A mixture of the enaminone (8c) (500 mg, 1.537 mmol) and Pd[O_2CMe]$_2$ (172 mg, 0.776 mmol) in chloroform (70 cm^3) was refluxed for 12 h. During the reaction yellow crystals were separated. Usual work-up and recrystallisation from chloroform afforded (9c) as yellow fine *needles* (463 mg, 80%), m.p. 243—245.5 °C (Found: C, 63.5; H, 6.0; N, 3.85. $C_{40}H_{44}N_2O_6Pd$ requires C, 63.6; H, 5.85; N, 3.7%); $\nu_{max.}$ at 1 590m and 1 570w cm^{-1}.

TABLE 2

Bond distances (Å) and angles (°) for complex (4a)

(*a*) Distances

Pd–O	1.972(7)	C(4)–C(5)	1.524(18)
Pd–N	2.030(8)	C(6)–C(7)	1.353(14)
Pd–O′	1.972(7)	C(6)–C(11)	1.385(13)
Pd–N′	2.030(8)	C(7)–C(8)	1.424(17)
Br–C(7)	1.900(10)	C(8)–C(9)	1.384(16)
O–C(2)	1.276(12)	C(9)–C(10)	1.404(19)
N–C(4)	1.330(12)	C(10)–C(11)	1.419(18)
N–C(6)	1.454(13)	$C_B(1)$–$C_B(2)$	1.417(22)
C(1)–C(2)	1.522(18)	$C_B(1)$–$C_B(3')$	1.364(23)
C(2)–C(3)	1.369(16)	$C_B(2)$–$C_B(3)$	1.378(22)
C(3)–C(4)	1.407(17)		

(*b*) Angles

O–Pd–N	92.1(3)	C(3)–C(4)–C(5)	118.5(10)
O–Pd–N′	87.9(3)	N–C(6)–C(7)	121.8(8)
N–Pd–O′	87.9(3)	N–C(6)–C(11)	118.7(8)
N–Pd–N′	180.0	C(7)–C(6)–C(11)	119.6(9)
N′–Pd–O′	92.1(3)	C(6)–C(7)–C(8)	122.2(9)
Pd–O–C(2)	126.5(7)	Br–C(7)–C(6)	120.8(7)
Pd–N–C(4)	124.2(7)	Br–C(7)–C(8)	117.0(8)
Pd–N–C(6)	117.3(6)	C(7)–C(8)–C(9)	118.8(11)
C(4)–N–C(6)	118.4(8)	C(8)–C(9)–C(10)	119.6(12)
O–C(2)–C(1)	114.9(9)	C(9)–C(10)–C(11)	119.9(11)
O–C(2)–C(3)	125.1(10)	C(6)–C(11)–C(10)	120.0(10)
C(1)–C(2)–C(3)	119.7(10)	$C_B(2)$–$C_B(1)$–$C_B(3')$	118.9(15)
C(2)–C(3)–C(4)	128.6(11)	$C_B(1)$–$C_B(2)$–$C_B(3)$	117.1(15)
N–C(4)–C(3)	122.9(9)	$C_B(2)$–$C_B(3)$–$C_B(1')$	123.9(16)
N–C(4)–C(5)	118.6(10)		

Crystal Structure Determination of (4a).—*Crystal data.* $C_{22}H_{22}Br_2N_2O_2Pd\cdot C_6H_6$, $M = 690.77$, Triclinic, space group $P\bar{1}$, $a = 8.034(5)$, $b = 11.613(5)$, $c = 7.637(10)$ Å, $\alpha = 98.67(5)$, $\beta = 88.44(5)$, $\gamma = 100.22(10)$°, $U = 693.2$ Å^3, $F(000) = 342$, $D_c = 1.655$ g cm^{-3}, $Z = 1$, Mo-K_α radiation $\lambda = 0.710\ 69$ Å, μ(Mo-K_α) = 35.5 cm^{-1}.

Structure analysis. The crystals of [Pd{N(C_6H_4Br-*o*)C(Me)CHC(Me)O}$_2$]·C_6H_6 were grown from a benzene solution as fine yellow needles. The specimen for *X*-ray diffraction study was sealed in a glass capillary to prevent the loss of solvent of crystallization. The lattice constants and intensity data were measured on a Philips four-circle *X*-ray diffractometer using Mo-K_α radiation monochromated by a graphite plate. The intensities of 2 951 independent reflections were measured within the 2θ angle of 40° by the θ—2θ scan method with a scan speed of 6° min^{-1} in θ. The size of the crystal was about 0.5 × 0.1 × 0.03 mm and no absorption correction was applied since the maximum μR value was 0.89. The crystal structure was solved by the heavy-atom method and refined by the least-squares method with block-diagonal approximations. The final *R* value was 0.092 including anisotropic thermal parameters. No hydrogen-atom contributions were taken into account.

TABLE 3

Deviations (Å) of atoms from the least-squares plane through the two chelate rings

Atom	Deviation	Atom	Deviation
Pd	0.000	O′	−0.069(7)
O	0.069(7)	C(2)′	0.021(8)
C(2)	−0.021(8)	C(3)′	0.039(8)
C(3)	−0.039(8)	C(4)′	−0.014(8)
C(4)	0.014(8)	N′	−0.036(7)
N	0.036(7)		

Tables of observed and calculated structure factors and anisotropic thermal parameters are in Supplementary Publication No. SUP 23143 (14 pp.).*

[1/102 *Received, 23rd January*, 1981]

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* For details see Notices to Authors No. 7, *J. Chem. Soc., Dalton Trans.*, 1980, Index issue.

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