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1,2-Bis-*N*-[2′-(diphenylphosphanyl)benzoyl]diaminobenzene, a New Chelating Ligand with Versatile Coordination Properties

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1,2-Bis-*N*-[2′-(diphenylphosphanyl)benzoyl]diaminobenzene (dppbH; **1**) was prepared by peptidic coupling and shown to exist, in the solid state, in the form of hydrogen-bonded dimers by single-crystal X-ray structure analysis. As expected, **1** reacts with [MCl_2(cod)] (M = Pd, Pt; cod = cyclooctadiene) to form square-planar complexes. However, in the case of palladium [$PdCl_2$(dppbH)] (**2**) is obtained, while in the case of platinum [Pt(dppb)] (**3**) is formed. Thus, the nature of the metal induces a completely different coordination mode: In **2**, the dppbH ligand only coordinates through the two phosphorus atoms, while in **3** a dppb ligand, formed by deprotonation of the two amino functions in dppbH, coordinates through the two phosphorus atoms and through the two nitrogen atoms. The single-crystal X-ray analyses of the two square-planar complexes reveal **2** to contain the two phosphorus atoms in a *trans* coordination mode and **3** to contain the two phosphorus atoms in a *cis* coordination mode.

Introduction

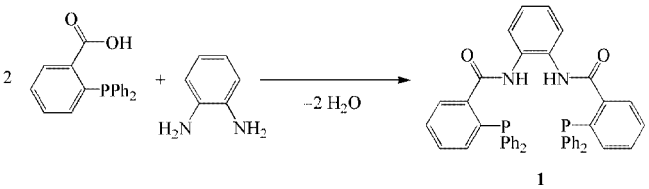
The synthesis of new, highly active complexes for homogeneously catalyzed processes is a challenge in coordination chemistry. Square-planar complexes of Pd^{II} and, to a minor extent, Pt^{II} have long been recognized to play a central role in catalytic reactions such as the classical Wacker process[1] and, more recently, in the co-polymerization of carbon monoxide and olefins.[2] Pd^{II} and Pt^{II} complexes are used in organic synthesis reactions such as Diels−Alder cycloadditions,[3] Baeyer−Villiger oxidation of ketones,[4] hydrogenolysis[5] or Sonogashira cross-coupling.[6]

Electron-rich ligands have been used to increase the electron density at the metal center in order to facilitate the oxidative addition step in a catalytic cycle.[7] Phosphane ligands have been extensively studied because of their electron-donating power. Diphosphane ligands have received special attention because they form, in general, more-stable (chelate) complexes than monophosphane ligands under catalytic conditions.[8] In the co-polymerization of olefins with carbon monoxide, Dosset et al.[7] showed that the catalytic activity of diphosphanylpalladium(II) complexes of the type [$PdCl_2\{Ar_2P(CH_2)_nPAr_2\}$] ($n$ = 1−3) depends upon the length of the carbon chain and upon the nature of the aryl groups. The six-membered chelate (n = 3) is an order of magnitude more active than the five-membered analogue (n = 2), while the four-membered chelate (n = 1) is essentially inactive. Complexes containing an *ortho* substituent at the aryl groups have a higher catalytic potential than those with *para* and *meta* substituents. It has been suggested that mononuclear *trans* bidentate complexes are more stable with large metallacycles than with medium-size metallacycles due to the increased flexibility of the larger ring size.[9] In general, the stability of the *trans* monomer increases with increasing chain length and reaches a maximum for a metallacycle containing 15 ring members.[10]

Herein, we report the synthesis of a new diphosphane ligand containing an *ortho*-phenylene spacer group for the synthesis of *trans*-dicoordinate as well as *cis*-tetracoordinate square-planar palladium(II) and platinum(II) complexes.

Results and Discussion

2-(Diphenylphosphanyl)benzoic acid, easily available from Wurtz coupling of sodium 2-chlorobenzoate and sodium diphenylphosphide,[11] is an attractive building block for the synthesis of diphosphane ligands by condensation of the acid function with diols, diamines or amino alcohols.[12] Peptidic coupling between *ortho*-diaminobenzene and two equivalents of 2-(diphenylphosphanyl)benzoic acid affords



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Scheme 1

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1,2-bis-*N*-[2′-(diphenylphosphanyl)benzoyl]diaminobenzene (dppbH; **1**) as a microcrystalline powder in good yield (Scheme 1).

The diphosphane ligand **1** is symmetrical and shows only one resonance in the $^{31}P\{^1H\}$ NMR spectrum at $\delta = -8.83$ ppm. In the ^1H NMR spectrum, **1** gives rise to a singlet at $\delta = 8.63$ ppm (in $CDCl_3$) corresponding to the amido protons. The amido function is also easily identified in the IR spectrum by two absorptions at 3324 cm^{-1} (ν_{NH}) and 1640 cm^{-1} (ν_{CO}). The molecular structure of **1**, established by single-crystal X-ray structure analysis, is shown in Figure 1.

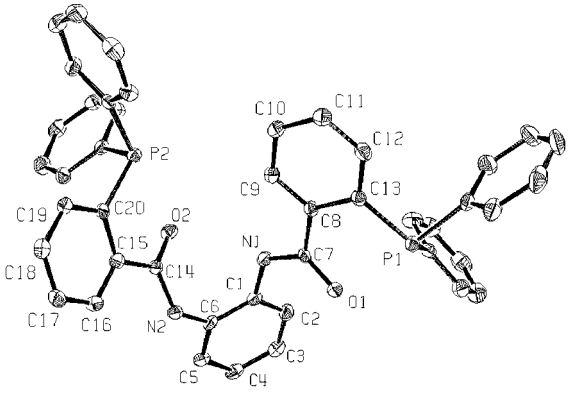


Figure 1. ORTEP view of **1**, displacement ellipsoids are drawn at the 50% probability level, hydrogen atoms are omitted for clarity; selected bond lengths (Å) and angles (°): C(7)−O(1) 1.237(3), C(14)−O(2) 1.235(3), C(7)−N(1) 1.348(4), C(14)−N(2) 1.355(4), C(13)−P(1) 1.847(3), C(20)−P(2) 1.853(3); O(1)−C(7)−N(1) 121.6(3), O(2)−C(14)−N(2) 121.8(3)

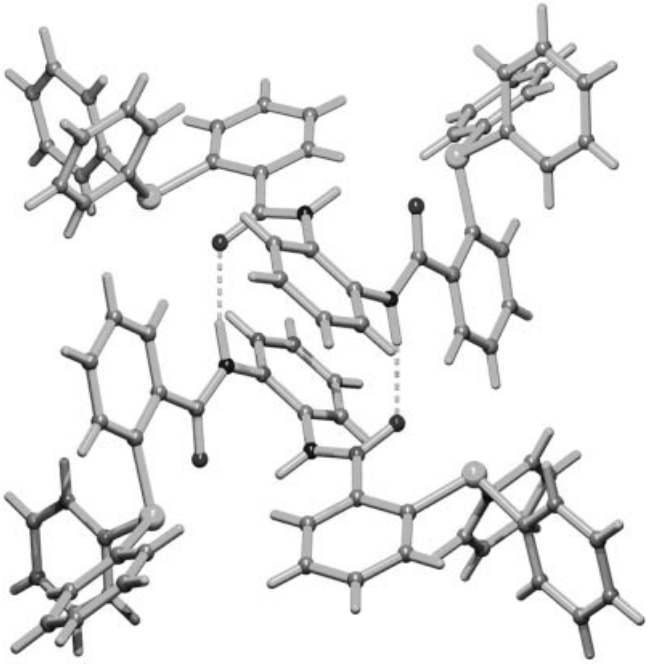
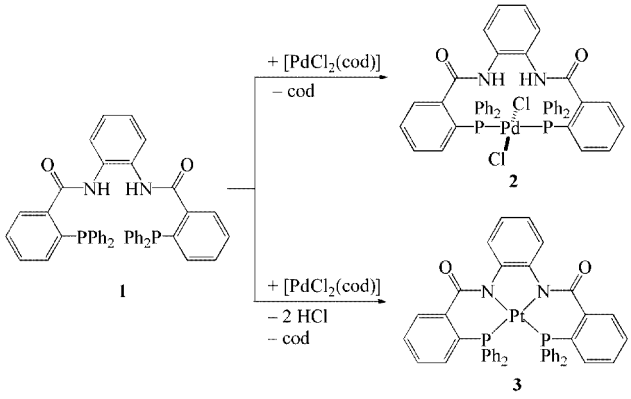


Figure 2. Dimeric structure of compound **1**

The crystal structure analysis reveals **1** to exist as a dimer in the solid state, thanks to hydrogen bonding between an NH and a CO function (see Figure 2). The N−O distance of the hydrogen bond [N(2)−H···O(1)] is 2.867(3) Å, with an N−H···O angle of 164.6°. The intramolecular distance between the two phosphorus atoms is 7.794(1) Å. In the crystal structure, there is no significant interaction between compound **1** and the dichloromethane molecule.

One equivalent of dppbH (**1**) reacts with [MCl_2(cod)] (M = Pd, Pt; cod = cyclooctadiene) to afford the corresponding complexes [$PdCl_2$(dppbH)] (**2**) and [Pt(dppb)] (**3**) in good yield (Scheme 2). The reaction was carried out in dilute solution to avoid the formation of di- or polynuclear species as observed for other diphosphane ligands.[13]



Scheme 2

[$PdCl_2$(dppbH)] (**2**) gives only one singlet at $\delta = 24.03$ ppm in the $^{31}P\{^1H\}$ spectrum, which is indicative of the coordination of both phosphorus atoms to the metal. The mass spectrum is in accordance with the presence of a mononuclear species containing two chloride ligands. The IR spectrum shows two bands at 3324 (ν_{NH}) and 1640 cm^{-1} (ν_{CO}) assigned to the amido functions.

The molecular structure of **2** shows the palladium atom to be in a square-planar geometry, surrounded by two chlorines and two phosphorus atoms (Figure 3). The chelating diphosphane ligand adopts a *trans* coordination geometry. The Cl−Pd−Cl as well as the P−Pd−P axes are almost linear, the corresponding angles being 177.03(10)° and 178.40(9)°, respectively. The two Pd−Cl distances are equivalent [2.310(2) and 2.311(2) Å], but interestingly one Pd−P distance is slightly longer (by 0.042 Å) than the other one. There is no meaningful interaction in the apical position of the palladium atom, the shortest distance to another atom being 2.96 Å [Pd(1)−O(2)]. In the crystal structure, only one chloroform molecule interacts with complex **2**, forming a weak hydrogen bond with one of the chlorine atoms, the Cl(2)−C(1S) distance being 3.49(1) Å and the C(1S)−H···Cl(2) angle being 150.3(1)°.

The formation of **3** is best monitored by $^{31}P\{^1H\}$ spectroscopy, the complex showing a triplet centered at $\delta = 6.20$ ppm due to the coupling between the two equivalent phosphorus atoms and the platinum atom (coupling con-

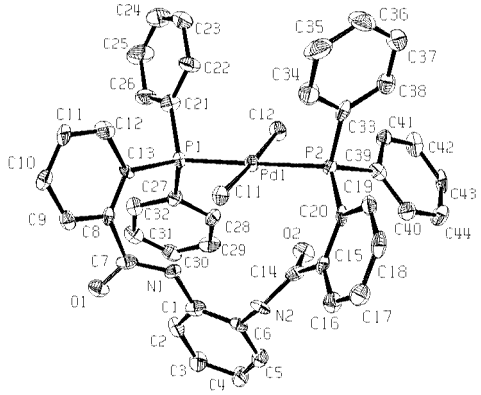


Figure 3. ORTEP view of complex **2**, displacement ellipsoids are drawn at the 50% probability level, hydrogen atoms and solvent molecules are omitted for clarity; selected bond lengths (Å) and angles (°): P(1)−Pd(1) 2.318(2), P(2)−Pd(1) 2.360(2), Cl(1)−Pd(1) 2.310(2), Cl(2)−Pd(1) 2.311(2), C(7)−O(1) 1.232(9), C(14)−O(2) 1.222(8), C(7)−N(1) 1.336(9), C(14)−N(2) 1.362(9), C(13)−P(1) 1.859(7), C(20)−P(2) 1.832(7); Cl(1)−Pd(1)−Cl(2) 177.03(10), Cl(1)−Pd(1)−P(1) 91.91(7), Cl(2)−Pd(1)−P(1) 86.27(8), Cl(1)−Pd(1)−P(2) 89.67(8), Cl(2)−Pd(1)−P(2) 92.17(8), P(1)−Pd(1)−P(2) 178.40(9), O(1)−C(7)−N(1) 123.2(7), O(2)−C(14)−N(2) 121.6(7)

stant 3120 Hz). IR spectroscopy shows that there is no ν_{NH} absorption (around 3300 cm^{-1}) any more, but a strong ν_{CO} band at 1602 cm^{-1} showing a strong bathochromic effect. It appears that the formation of **3** is accompanied by the loss of two equivalents of HCl. Unlike $[Pt\{Ph_2PC_6H_4NC(O)C_6H_4\}_2]$,[14] complex **3** does not give the dichloroplatinum complex $[PtCl_2(dppbH)]$ in the presence of HCl.

The molecular structure of **3** shows the platinum atom in a distorted square-planar geometry (Figure 4). Unlike in **2**, the chelating diphosphane ligand adopts a *cis* coordination geometry, with the two nitrogen atoms also coordinating to the metal. The formation of five- and six-membered chelate rings imposes a considerable distortion around the platinum atom. The N−Pt−P angles [83.47(16) and 89.71(16)°] are slightly acute due to the bidentate $PC_6H_4C(O)N$ units. The atoms Pt(1), P(1), P(2), N(1) and N(2) are almost coplanar, with an average deviation of 0.0674 Å; the metal lies out of the plane by 0.078(2) Å. In the crystal structure, there is no meaningful interaction between complex **3** and the chloroform molecule.

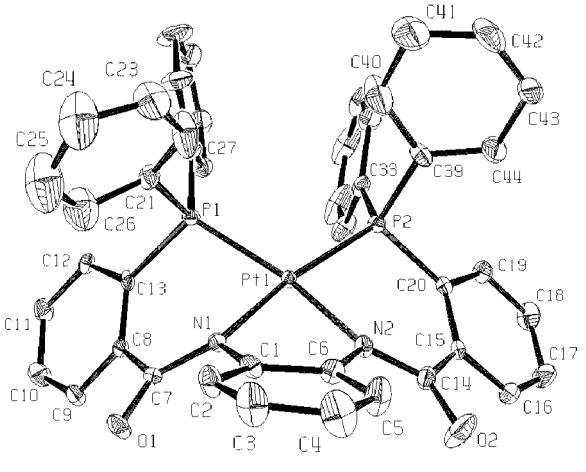


Figure 4. ORTEP view of complex **3**, displacement ellipsoids are drawn at the 35% probability level, hydrogen atoms and solvent molecules are omitted for clarity; selected bond lengths (Å) and angles (°): P(1)−Pt(1) 2.243(2), P(2)−Pt(1) 2.234(2), N(1)−Pt(1) 2.035(6), N(2)−Pt(1) 2.067(5), C(7)−O(1) 1.215(8), C(14)−O(2) 1.240(9), C(7)−N(1) 1.367(8), C(14)−N(2) 1.356(10), C(13)−P(1) 1.830(6), C(20)−P(2) 1.820(7); N(1)−Pt(1)−N(2) 80.3(2), N(1)−Pt(1)−P(1) 83.47(16), N(2)−Pt(1)−P(1) 161.74(18), N(1)−Pt(1)−P(2) 169.92(15), N(2)−Pt(1)−P(2) 89.71(16), P(1)−Pt(1)−P(2) 106.26(6), O(1)−C(7)−N(1) 125.2(6), O(2)−C(14)−N(2) 122.8(7)

The stability of complex **2** towards base attack and that of complex **3** towards acid attack has been studied in order to check the possibility of converting these *cis* or *trans* complexes into their corresponding deprotonated or protonated species. However, complex **2** is not deprotonated by an excess of triethylamine to give [Pd(dppb)], the $^{31}P\{^1H\}$ NMR signal of **2** being unchanged in $CDCl_3$ even after 24 hours in the presence of NEt_3 (100 equiv.). Likewise, complex **3** is not protonated by concentrated hydrochloric acid to give $[PtCl_2(dppbH)]$, the $^{31}P\{^1H\}$ NMR signal of **3** being unchanged in $CDCl_3$ even after 24 hours in the presence of HCl (100 equiv.).

Conclusion

We have synthesized a new diphosphane ligand, 1,2-bis-*N*-[2′-(diphenylphosphanyl)benzoyl]diaminobenzene (dppbH; **1**), which shows a different coordination pattern towards palladium(II) and platinum(II): it reacts with $[MCl_2(cod)]$ (M = Pd, Pt; cod = cyclooctadiene) to form the square-planar complexes $[PdCl_2(dppbH)]$ (**2**) and [Pt(dppb)] (**3**), respectively. In **2**, the dppbH ligand is coordinated by the two phosphorus atoms in a *trans* configuration, while in **3** the two phosphorus atoms are coordinated in a *cis* configuration, the two nitrogen atoms of the dppb ligand being coordinated as well.

Experimental Section

General Remarks: Dichloromethane was dried and distilled under nitrogen prior to use. All reactions were carried out under nitrogen, using standard Schlenk techniques. All other reagents were purchased (Aldrich, Fluka) and used as received. NMR spectra were recorded using a Varian Gemini 200BB instrument and referenced to the signals of the residual protons in the deuterated solvents. Electrospray mass spectra were obtained in positive-ion mode with an LCQ Finnigan mass spectrometer. IR spectra were recorded with a Perkin−Elmer Spectrum One FTIR spectrometer. Microanalyses were carried out by the Laboratory of Pharmaceutical Chemistry, University of Geneva, Switzerland.

Preparation of 1,2-Bis-*N*-[2′-(diphenylphosphanyl)benzoyl]diaminobenzene (dppbH; 1): A solution of 2-diphenylphosphanylbenzoic acid (1.0 g, 3.26 mmol), *N*,*N*-dicyclohexylcarbodiimide (2.7 g, 13.05 mmol), 4-(dimethylamino)pyridine (100 mg, 0.82 mmol), 4-pyrrolidinopyridine (100 mg, 0.68 mmol), and 1,2-diaminobenzene (175 mg, 1.62 mmol) in CH_2Cl_2 (40 mL) was allowed to stand at

room temperature under nitrogen, until the esterification was complete. The resulting solution was concentrated and filtered three times to remove *N*,*N*-dicyclohexyl urea. The filtrate was then concentrated under reduced pressure. The residue was submitted to column chromatography on silica, eluting with hexane/acetone (3:1). The product was isolated from the third fraction by the evaporation of the solvent, giving **1** (550 mg, 0.81 mmol; 50%) as a white solid. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 8.63 (s, 2 H, NH), 7.5−6.9 (m, 32 H, ArH) ppm. $^{13}C\{^1H\}$ NMR (50 MHz, $CDCl_3$): δ = 168.12, 143.45, 141.20, 141.00, 140.50, 137.62, 137.39, 134.62, 133.90, 130.84, 129.68, 128.94, 128.80, 128.65 ppm. $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$): δ = −8.83 ppm. IR (KBr): $\tilde{\nu}$ = 3324 m (N−H amide), 1640 s (C=O amide) cm^{-1}. ESI-MS: *m*/*z* = 685 [M + H$^+$]. $C_{44}H_{34}N_2O_2P_2$ (684.21): calcd. C 77.2, H 5.0, N 4.1; found C 76.8, H 4.8, N 4.3.

Preparation of [$PdCl_2$(dppbH)] (2): A solution of [$PdCl_2$(cod)] (42 mg, 0.15 mmol) and **1** (100 mg, 0.15 mmol) in CH_2Cl_2 (20 mL) was allowed to stand for 3 hours at room temperature and then filtered. The filtrate was concentrated under reduced pressure. The residue was submitted to column chromatography on silica, eluting with dichloromethane/ethanol (4:1). The product was isolated from the first fraction by the evaporation of the solvent, giving **2** (95 mg, 0.11 mmol; 79%) as a yellow-orange solid. 1H NMR (200 MHz, $CDCl_3$): δ = 8.66 (s, 2 H, NH), 8.1−6.9 (m, 32 H, ArH) ppm. $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); δ = 24.03 ppm. IR (KBr): $\tilde{\nu}$ = 3302 m (N−H amide), 1662 s (C=O amide) cm^{-1}. ESI-MS: *m*/*z* = 885 [M + Na$^+$]. $C_{44}H_{34}Cl_2N_2O_2P_2Pd$ (861.51): calcd. C 61.3, H 4.0, N 3.3; found C 61.1, H 4.1, N 3.2.

Preparation of [Pt(dppb)] (3): A solution of [$PtCl_2$(cod)] (55 mg, 0.15 mmol) and **1** (100 mg, 0.15 mmol) in CH_2Cl_2 (20 mL) was allowed to stand for two hours at room temperature and then filtered. The filtrate was concentrated under reduced pressure. The residue was submitted to column chromatography on silica, eluting with dichloromethane/ethanol (3:1). The product was isolated from the first fraction by the evaporation of the solvent, giving **3** (80 mg, 0.09 mmol; 61%) as a yellow-green solid. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.9−6.7 (m, 32 H, ArH) ppm. $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); δ = 6.20 (t, $^1J_{Pt,P}$ = 3120 Hz) ppm. IR (KBr): $\tilde{\nu}$ = 1602 s (C=O amide) cm^{-1}. ESI-MS: *m*/*z* = 878 [M + H$^+$], 900 [M + Na$^+$]. $C_{44}H_{32}N_2O_2P_2Pt$ (877.28): calcd. C 60.2, H 3.7, N 3.2; found C 60.0, H 3.6, N 3.4.

X-ray Crystallographic Study

1·CH_2Cl_2: $C_{45}H_{36}Cl_2N_2O_2P_2$, *M* = 769.60 g·mol^{-1}, triclinic, $P\bar{1}$ (no. 2), *a* = 9.2473(8), *b* = 14.0068(14), *c* = 14.5320(16) Å, α = 89.904(12), β = 89.630(12), γ = 89.693(11)°, *U* = 1882.2(3) Å^3, *T* = 153 K, *Z* = 2, μ(Mo-K_α) = 0.300 mm^{-1}, 12481 reflections measured, 6853 unique (R_{int} = 0.0607) which were used in all calculations. The final *wR* (F^2) was 0.1126 (all data).

2·2$CHCl_3$: $C_{46}H_{36}Cl_8N_2O_2P_2Pd$, *M* = 1100.71 g·mol^{-1}, triclinic, $P\bar{1}$ (no. 2), *a* = 9.9013(11), *b* = 15.1734(18), *c* = 16.7870(18) Å, α = 78.059(14), β = 76.629(13), γ = 78.630(14)°, *U* = 2370.8(5) Å^3, *T* = 153 K, *Z* = 2, μ(Mo-K_α) = 0.950 mm^{-1}, 17568 reflections measured, 8657 unique (R_{int} = 0.1286) which were used in all calculations. The final *wR*(F^2) was 0.1015 (all data).

3·$CHCl_3$: $C_{45}H_{33}Cl_3N_2O_2P_2Pt$, *M* = 997.11 g·mol^{-1}, monoclinic, $P2_1/n$ (no. 14), *a* = 12.0140(17), *b* = 11.259(3), *c* = 28.720(4) Å, β = 95.026(16)°, *U* = 3870.0(13) Å^3, *T* = 153 K, *Z* = 4, μ(Mo-K_α) = 3.958 mm^{-1}, 20851 reflections measured, 7307 unique (R_{int} = 0.0544) which were used in all calculations. The final *wR*(F^2) was 0.0984 (all data).

The data were measured using a Stoe Image Plate Diffraction system equipped with a φ circle, using Mo-K_α graphite-monochromated radiation (λ = 0.71073 Å) with φ range 0−200°, increment of 1.5, 1.4 and 0.8° respectively, 2θ range from 2.0−26°, $D_{max}-D_{min}$ = 12.45−0.81 Å. The structures were solved by direct methods using the program SHELXS-97.[15] The refinement and all further calculations were carried out using SHELXL-97.[16] The H-atoms were included in calculated positions and treated as riding atoms using the SHELXL default parameters. The non-H atoms were refined anisotropically, using weighted full-matrix least-square on F^2. Figures were drawn with ORTEP.[17]

CCDC-212037 (**1**), -212039 (**2**), and -212038 (**3**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

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