Polyhedron 57 (2013) 20–23

Contents lists available at SciVerse ScienceDirect

Polyhedron

journal homepage: www.elsevier.com/locate/poly

Palladium(II)-assisted [2+3] cycloaddition of nitrones to organonitriles: Synthesis of 2,3-dihydropyrrolo[1,2-a]quinazolin-5-one and Δ^4-1,2,4-oxadiazoline derivatives

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ARTICLE INFO

Article history:
Received 11 February 2013
Accepted 4 April 2013
Available online 16 April 2013

Keywords:
Palladium(II) chloride
Nitrile
Nitrone
[2+3]Cycloaddition
One-pot synthesis
Dppe

ABSTRACT

The [2+3] cycloadditions of different organonitriles NCR ($R^1 = C_6F_5$, R^2 = Pr, R^3 = *p*-$CHOC_6H_4$, R^4 = Ph) with the cyclic nitrone $^{-}O^{+}N{=}CHCH_2CH_2CMe_2$ or acyclic nitrone $^{-}O^{+}N(Me){=}C(H)(C_6H_2Me_3$-2,4,6), in the presence of $PdCl_2$, give the corresponding fused tricyclic fluorinated ketoimine *trans*-$[PdCl_2\{NC(=O)C_6F_4NCCH_2CH_2CMe_2\}_2]$ (**1**) (**1**), fused bicyclic Δ^4-1,2,4-oxadiazoline *trans*-$[PdCl_2\{N{=}C(R)ONCHCH_2CH_2CMe_2\}_2]$ (R^2 = Pr (**2**), R^3 = *p*-$CHOC_6H_4$ (**3**)) or monocyclic Δ^4-1,2,4-oxadiazoline *trans*-$[PdCl_2\{N{=}C(Ph)ON(Me)C(H)(C_6H_2Me_3$-2,4,6)$\}_2]$ (**4**) (**4**) palladium(II) complexes. The *free* tetrafluoro-2,3-dihydropyrrolo[1,2-a]quinazolin-5-one (**1a**) and Δ^4-1,2,4-oxadiazolines (**2a**–**4a**) are liberated upon reaction of complexes **1**–**4** with a diphosphine (dppe). All the compounds were characterized by elemental analyses, ESI^+-MS, IR, ^1H and ^{13}C NMR spectroscopies.

1. Introduction

Oxadiazolines represent an important class of heterocycles which, although known for over a century, are rather limited in number. In recent years, these heterocycles have drawn greater attention due to their promising biological activities [1]. Also fluorine containing heterocycles have gained the attention of chemists and biologists because many derivatives show a high biological potency in their ability to inhibit specific enzymes, their good solubility in lipids and their ability to penetrate through cell membranes [2].

The [2+3] cycloaddition of nitrones to organonitriles, NCR, is one of the most important routes for the synthesis of 1,2,4-oxadiazolines. However, there are some limitations for this method, as only organonitriles activated with a strong electron-withdrawing group (*e.g.* $R^1 = CCl_3$, CO_2R^2) directly ligated to the triple bond can react with the nitrones. Moreover, rather harsh conditions and/or long reaction times are required [3]. Therefore, the development of a new approach to accelerate the [2+3] cycloaddition of nitrones with nitriles under milder conditions is an important aim of the research in this area. In this context, the coordination of nitriles to a suitable metal centre became a widespread synthetic strategy and facile route to the synthesis of a large variety of compounds, inaccessible directly by pure organic chemistry [4–8]. The increased coupling ability of nitriles with different types of nucleophiles [9,10] or 1,3-dipole reagents [11,12] when coordinated to a metal centre has already been well demonstrated.

In our current work, the main objectives were (i) to prepare palladium(II) complexes with *N*-heterocyclic ligands, and (ii) to investigate the liberation of those heterocycles from their respective metal complexes.

2. Results and discussion

2.1. Syntheses of palladium(II) complexes

Our work started with the [2+3] cycloadditions of different organonitriles, NCR ($R^1 = C_6F_5$, R^2 = Pr, R^3 = *p*-$CHOC_6H_4$, R^4 = Ph), with pyrroline *N*-oxide (cyclic nitrone) $^{-}O^{+}N{=}CHCH_2CH_2CMe_2$ or acyclic nitrone $^{-}O^{+}N(Me){=}C(H)(C_6H_2Me_3$-2,4,6), in the presence of $PdCl_2$, to give the corresponding fused tricyclic fluorinated ketoimine *trans*-$[PdCl_2\{NC(=O)C_6F_4NCCH_2CH_2CMe_2\}_2]$ (**1**) (**1**), fused bicyclic Δ^4-1,2,4-oxadiazoline *trans*-*trans*-$[PdCl_2\{N{=}C(R)ONCHCH_2CH_2CMe_2\}_2]$ (R^2 = Pr (**2**), R^3 = *p*-$CHOC_6H_4$ (**3**)) or monocyclic Δ^4-1,2,4-oxadiazoline $[PdCl_2\{N{=}C(Ph)ON(Me)C(H)(C_6H_2Me_3$-2,4,6)$\}_2]$ (**4**) (**4**) palladium(II) complexes (Scheme 1).

Complexes **1** and **2** have been reported previously by us [12a]. The new Δ^4-1,2,4-oxadiazoline palladium(II) complexes **3** and **4** were characterized by elemental analyses, ESI^+-MS, IR, ^1H and ^{13}C NMR spectroscopies (see Section 4). In the IR spectra of **3** and **4**,

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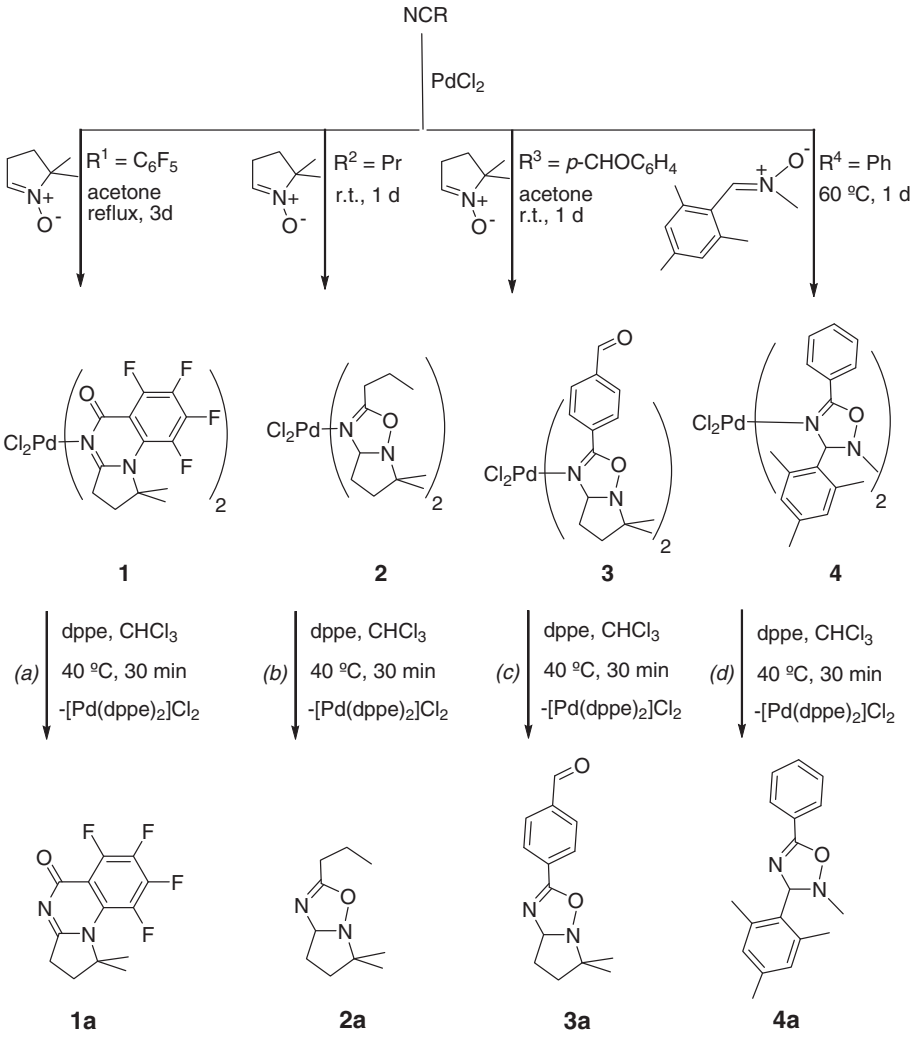
http://dx.doi.org/10.1016/j.poly.2013.04.017



Scheme 1.

ν(C═N) is detected as a strong band in the 1639–1657 cm^{-1} range, which replaces the higher wavenumber ν(C≡N) band of the starting organonitriles NCR. The NMR results confirm the formation of the oxadiazoline ring [11a,12a,b]. Hence in the ^1H NMR spectra of **3** and **4**, for example, the expected signals of the N–C*H*–N protons are detected in the 5.66–6.15 ppm range, whereas in the ^{13}C NMR spectra, the N–*C*H–N resonances appear in the 89.5–91.3 ppm range and the *C*═N resonances are detected in the 163.6–163.7 ppm range.

2.2. Liberation of the heterocyclic compounds *1a–4a*

The liberation of the *N*-heterocyclic ligands **1a**–**4a** was carried out by the reaction of complexes **1**–**4** with 2 eq of 1,2-bis(diphenylphosphino)ethane (dppe). When the precipitation of the colourless compound $[Pd(dppe)_2]Cl_2$ (identified by IR and $^{31}P\{^1H\}$ NMR [13]) was complete, the formation of the free heterocycles tetrafluoro-2,3-dihydropyrrolo[1,2-*a*]quinazolin-5-one (**1a**) (Scheme 1, reaction *a*) and Δ^4-1,2,4-oxadiazolines (**2a**–**4a**) (Scheme 1, reactions *b*–*d*) was confirmed by elemental analyses, ESI^+-MS, IR, ^1H and ^{13}C NMR spectroscopies (see Section 4). For example, the IR spectrum of **1a** exhibits strong ν(NC═O) and ν(C═N) vibrations at 1676 and 1583 cm^{-1} (1691 and 1649 cm^{-1} for complex **1** [12a]), respectively. In the ^1H NMR spectrum of **1a** the resonances of the methyl and methylene groups are observed at δ 1.75, 1.76, 2.27 and 3.15 (δ 1.78, 1.79, 2.46 and 4.61 for complex **1**). Moreover, in the ^{13}C NMR spectrum of **1a** the resonances at 151.5 and 167.4 ppm are assigned to the N═*C*N and N*C*═O moieties (159.1 and 169.8 ppm for complex **1** [12a]), respectively.

3. Conclusions

In this work we have shown that $PdCl_2$ activates very effectively organonitriles, NCR, towards coupling with nitrones (cyclic and acyclic ones) to give, under mild conditions and *via* a single-pot [2+3] cycloadditions, fused tricyclic fluorinated ketoimine, fused bicyclic or monocyclic Δ^4-1,2,4-oxadiazoline palladium(II) complexes, in moderate to good yields. In addition, the *N*-heterocyclic ligands can be liberated from the metal centre and the current method provides a more facile and cheaper one-pot palladium(II)-mediated route to the syntheses of *free* tetrafluoro-2,3-dihydropyrrolo[1,2-*a*]quinazolin-5-one and Δ^4-1,2,4-oxadiazoline derivatives, in comparison with related platinum systems which do not proceed *via* single-pot reactions [11c,f].

4. Experimental

4.1. General

Solvents and reagents were obtained from commercial sources (Aldrich) and used as received. Acyclic nitrone was prepared according to the published methods [11a,14]. Complexes **1** and **2**

have been reported previously by us [12a]. C, H and N elemental analyses were carried out by the Microanalytical Service of the Instituto Superior Técnico. ^{1}H and ^{13}C spectra (in $CDCl_3$) were measured on a Bruker Avance II 400 MHz (UltraShield™ Magnet) spectrometer at ambient temperature. ^{1}H and ^{13}C chemical shifts (δ) are expressed in ppm relative to $Si(Me)_4$. *J* values are in Hz. Infrared spectra (4000–400 cm^{-1}) were recorded on a Bio-Rad FTS 3000MX and a Jasco FT/IR-430 instrument in KBr pellets and the wavenumbers are in cm^{-1}. Electrospray mass spectra were carried out with an ion-trap instrument (Varian 500-MS LC Ion Trap Mass Spectrometer) equipped with an electrospray (ESI) ion source. The solutions in methanol were continuously introduced into the mass spectrometer source with a syringe pump at a flow rate of 10 μL/min. The drying gas temperature was maintained at 350 °C and dinitrogen was used as the nebulizer gas at a pressure of 35 psi. Scanning was performed from *m*/*z* = 50 to 1500.

4.2. Synthesis of complex **3**

A solution of 4-cyanobenzaldehyde (29.6 mg, 0.226 mmol) in acetone (4 mL) was added at room temperature to cyclic nitrone (25.6 mg, 0.226 mmol) and palladium(II) chloride (20.0 mg, 0.113 mmol), and the mixture was stirred at room temperature for 1 d. Along the course of the reaction, the brown powder of $PdCl_2$ dissolved, forming an homogeneous light yellow solution. The reaction mixture was then dried *in vacuo*, washed with three 5 mL portions of diethyl ether and dried under air. The final complex **3** was recrystallized from acetone.

***Trans*-[PdCl$_2${N=C(*p*-CHOC$_6$H$_4$)ONCHCH$_2$CH$_2$CMe$_2$}$_2$] 3**: yield, 79%. IR (cm^{-1}): 1639 *ν*(C=N) and 1705 *ν*(C=O). ^{1}H NMR ($CDCl_3$), *δ*: 1.30 and 1.32 (two s, 6H, two CH_3), 2.07 (t, J_{HH} 7.5 Hz, 2H, CH_2), 3.39 (t, J_{HH} 7.5 Hz, 2H, CH_2), 5.66 (dd, J_{HH} 7.2 Hz, J_{HH} 3.3 Hz, 1H, N–C*H*–N), 8.07 (d, J_{HH} 7.8 Hz, 2H, C$H_{aromatic}$), 8.93 (d, J_{HH} 7.8 Hz, 2H, C$H_{aromatic}$), 10.11 (s, 1H, C*H*O). ^{13}C{^{1}H} NMR ($CDCl_3$), *δ*: 21.9 and 27.6 (CH_3), 29.3 and 34.3 (CH_2), 69.9 (Me_2*C*–N), 89.5 (N–*C*H–N), 128.3, 130.6, 137.2 and 139.3 ($C_{aromatic}$), 163.7 (*C*(O)=N), 191.1 (HC=O). ESI$^+$-MS, *m*/*z*: 667 [M+H]$^+$. *Anal.* Calc. for $C_{28}H_{32}N_4O_4Cl_2Pd$: C, 50.50; H, 4.84; N, 8.41. Found: C, 50.39; H, 4.54; N, 8.45%.

4.3. Synthesis of complex **4**

A solution of acyclic nitrone (39.6 mg, 0.224 mmol) in benzonitrile (4 mL) was added at room temperature to palladium(II) chloride (20.0 mg, 0.113 mmol), and the mixture was heated at 60 °C under stirring for 1 d. The solvent was then removed *in vacuo* and the resulting oily residue was treated in a manner similar to that described above to obtain the final complex **4**.

[PdCl$_2${N=C(Ph)ON(Me)C(H)(C$_6$H$_2$Me$_3$-2,4,6)}$_2$] **4**.: yield, 67%. IR (cm^{-1}): 1657 (C=N). ^{1}H NMR ($CDCl_3$), *δ*: 2.32, 2.49 and 2.91 (three s, 9H, CH_3Ph), 3.17 (s, 3H, CH_3N), 6.15 (s, 1H, N–C*H*–N), 6.90–7.97 (m, 7H, C$H_{aromatic}$). ^{13}C{^{1}H} NMR ($CDCl_3$), *δ*: 20.1, 20.7 and 21.4 (*C*H$_3$Ph), 47.5 (CH_3N), 91.3 (N–*C*H–N), 128.5, 129.4, 131.7, 133.1, 133.8, 136.3, 137.9 and 140.3 ($C_{aromatic}$), 163.6 (C(O)=N). ESI$^+$-MS, *m*/*z*: 703 [M−Cl]$^+$. *Anal.* Calc. for $C_{36}H_{40}N_4O_2Cl_2Pd$: C, 58.58; H, 5.46; N, 7.59. Found: C, 58.63; H, 5.69; N, 7.66%.

4.4. Liberation of the new heterocycles **1a–4a** from complexes **1–4**

1,2-Bis(diphenylphosphino)ethane (dppe) (2 eq) was added to a solution of **1** (20.0 mg, 0.027 mmol), **2** (20.0 mg, 0.037 mmol), **3** (20.0 mg, 0.029 mmol) or **4** (20.0 mg, 0.028 mmol) in $CHCl_3$ (5 mL) and the mixture was allowed to stand at 40 °C for 30 min, whereupon the colourless [Pd(dppe)$_2$]Cl$_2$ precipitated. This precipitate was separated by filtration and identified by ^{31}P{^{1}H}($CDCl_3$) NMR: *δ* = 55.9 (56.7 ppm [13]). The solution was then dried *in vacuo*, washed with three 5 mL portions of hexane and dried under air.

No reaction was observed using complex **1** and 1 eq of dppe, and only the starting material was recovered.

NC(=O)C$_6$F$_4$NCCH$_2$CH$_2$CMe$_2$ 1a: : yield, 90%. IR (cm^{-1}): 1676 *ν*(NC=O), 1583 *ν*(NC=N). ^{1}H NMR ($CDCl_3$), *δ*: 1.75 and 1.76 (two s, 6H, two CH_3), 2.27 (t, J_{HH} 8 Hz, 2H, CH_2), 3.15 (t, J_{HH} 8 Hz, 2H, CH_2). ^{13}C{^{1}H} NMR ($CDCl_3$), *δ*: 27.1 and 27.3 (CH_3), 31.3 and 38.3 (CH_2), 71.6 (*C*(Me)$_2$), 126.9–134.4 ($C_{aromatic}$), 151.5 (NC=N), 167.4 (NC=O). ESI$^+$-MS, *m*/*z*: 287 [M + H]$^+$. *Anal.* Calc. for $C_{13}H_{10}N_2OF_4$: C, 54.55; H, 3.52; N, 9.79. Found: C, 54.73; H, 3.34; N, 9.54%.

N=C(Pr)ONCHCH$_2$CH$_2$CMe$_2$ 2a: : yield, 87%. IR (cm^{-1}): 1691 *ν*(C=N). ^{1}H NMR ($CDCl_3$), *δ*: 0.99 (t, J_{HH} 7.2 Hz, 3H, CH_3CH$_2$), 1.31 and 1.39 (two s, 6H, two CH_3), 1.62–1.72 (m, 2H, CH_2), 2.20–2.25 (m, 2H, CH_2), 2.90–2.92 (m, 4H, CH_2), 5.15 (m, 1H, N–C*H*–N).^{13}C{^{1}H} NMR ($CDCl_3$), *δ*: 14.2 (*C*H$_3$CH$_2$), 19.5 (*C*H$_2$CH$_3$), 22.5 and 27.9 ((*C*H$_3$)$_2$C), 29.7 and 31.1 (*C*H$_2$CH$_2$), 32.1 (*C*H$_2$CH$_2$CH$_3$), 61.7 (Me$_2$*C*–N), 87.1 (N–*C*H–N), 167.7 (*C*(O)=N). ESI$^+$-MS, *m*/*z*: 183 [M+H]$^+$. *Anal.* Calc. for $C_{10}H_{18}N_2O$: C, 65.90; H, 9.95; N, 15.37. Found: C, 65.50; H, 9.57; N, 15.55%.

N=C(*p*-CHOC$_6$H$_5$)ONCHCH$_2$CH$_2$CMe$_2$ 3a. : yield, 85%. IR (cm^{-1}): 1635 *ν*(C=N) and 1681 *ν*(C=O). ^{1}H NMR ($CDCl_3$), *δ*: 1.27 and 1.31 (two s, 6H, two CH_3), 1.96 (t, J_{HH} 7.5 Hz, 2H, CH_2), 2.44 (t, J_{HH} 7.5 Hz, 2H, CH_2), 5.17 (m, 1H, N–C*H*–N), 7.48 (d, J_{HH} 7.8 Hz, 2H, C$H_{aromatic}$), 7.89 (d, J_{HH} 7.8 Hz, 2H, C$H_{aromatic}$), 10.09 (s, 1H, C*H*O). ^{13}C{^{1}H} NMR ($CDCl_3$), *δ*: 22.1 and 28.2 (CH_3), 28.7 and 31.7 (CH_2), 68.2 (Me$_2$*C*–N), 90.5 (N–*C*H–N), 128.9, 130.7, 134.3 and 138.5 ($C_{aromatic}$), 159.9 (*C*(O)=N), 191.5 (HC=O). ESI$^+$-MS, *m*/*z*: 245 [M + H]$^+$ and 277 [M + Na]$^+$. *Anal.* Calc. for $C_{14}H_{16}N_2O_2$: C, 68.83; H, 6.60; N, 11.47. Found: C, 68.49; H, 6.54; N, 11.65%.

N=C(Ph)ON(Me)C(H)(C$_6$H$_2$Me$_3$-2,4,6) 4a. : yield, 80%. IR (cm^{-1}): 1675 (C=N). ^{1}H NMR ($CDCl_3$), *δ*: 2.18, 2.26 and 2.42 (three s, 9H, CH_3Ph), 2.52 (s, 3H, CH_3N), 5.45 (s, 1H, N–C*H*–N), 7.43–7.94 (m, 7H, C$H_{aromatic}$). ^{13}C{^{1}H} NMR ($CDCl_3$), *δ*: 20.3, 20.9 and 21.7 (*C*H$_3$-Ph), 47.1 (CH_3N), 90.7 (N–*C*H–N), 126.9, 127.4, 128.6, 131.8, 131.9, 133.9, 136.9 and 140.1 ($C_{aromatic}$), 161.7 (C(O)=N). ESI$^+$-MS, *m*/*z*: 281 [M+H]$^+$. *Anal.* Calc. for $C_{18}H_{20}N_2O$: C, 77.11; H, 7.19; N, 9.99. Found: C, 77.46; H, 7.34; N, 9.55%.

Acknowledgments

This work has been supported by the Fundação para a Ciência e a Tecnologia (FCT), Portugal. The author expresses gratitude to the FCT and the Instituto Superior Técnico (IST) for his research contract (Ciência 2007 program). J.L. gratefully acknowledges the Portuguese NMR Network (IST-UTL Center) for access to the NMR facility. Thanks are also due to Ms. Ana Dias for the analytical service.

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