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Authors: Phil Liebing and Frank T. Edelmann

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Trifluoromethylated 3-(Pyrazol-1-yl)-Propanamide (PPA) Ligands

Phil Liebing,* and Frank T. Edelmann*

Otto-von-Guericke-Universität Magdeburg, Chemisches Institut, Universitätsplatz 2, 39106 Magdeburg, Germany, phil.liebing@ovgu.de,
frank.edelmann@ovgu.de

Dedicated to Professor Antonio Togni on the occasion of his 65th birthday

The new PPA ligands 3-(3-trifluoromethyl-5-methylpyrazol-1-yl)-propanamide (CF_3MePPA ; **3**) and 3-(3,5-bis(trifluoromethyl)pyrazol-1-yl)-propanamide ($(\text{CF}_3)_2\text{PPA}$; **4**) were synthesized by Aza-Michael addition of the parent pyrazole derivatives (**1**, **2**) to acrylamide. Both products were characterized by elemental analyses, IR and NMR spectroscopy, and mass spectrometry. X-ray structure determination of **3** revealed the presence of a one-dimensional hydrogen-bonded structure in the solid state. The ligating ability of the new ligands towards PdCl_2 was studied, showing that **3** behaves similar to Me_2PPA and reacts cleanly with PdCl_2 to afford the sparingly soluble complex $\text{PdCl}_2(\text{CF}_3\text{MePPA-}\kappa\text{N})_2$ (**5**). By contrast, the donor ability of pyrazolyl group in **4** was found to be considerably reduced, thus resulting in the formation of the unusual complex $\text{PdCl}_2\{(\text{CF}_3)_2\text{PPA-}\kappa\text{N}\}\{(\text{CF}_3)_2\text{PPA-}\kappa\text{O}\}$ (**6**).

Keywords: pyrazole ligands • fluorinated ligands • palladium complexes • X-ray structure elucidation • hydrogen bonding

Introduction

Due to their potential applications in the field of homogeneous catalysis, there is a significant research interest in multifunctional ligands based on pyrazole.^[1–5] Moreover, polydentate pyrazole-based ligands have gained relevance as potential metalloprotein mimetics,^[6–8] and as building blocks for metal-organic frameworks.^[9–13] In the course of our ongoing research on ligands based on the 3-(pyrazol-1-yl)propanamide (PPA) scaffold, we reported various transition metal complexes, showing that their coordination chemistry is diverse.^[14–19] The most frequently observed coordination mode for first-row transition metals is $\kappa\text{N},\kappa\text{O}$ -chelation,^[14–18] involving the pyrazolyl-nitrogen and amide-oxygen donor atoms (Figure 1, a). In contrast, for the less oxophilic second-row transition metals $\text{Pd}^{[19]}$ and $\text{Ag}^{[15]}$ κN -monodentate coordination *via* the pyrazolyl group appeared to be most favored (Figure 1, b). Due to the presence of amide N-H moieties as a potent hydrogen-bond donor, the free ligands as well as the complexes are aggregated into infinite supramolecular structures by N-H...O, N-H...N, or N-H...Cl bonds.

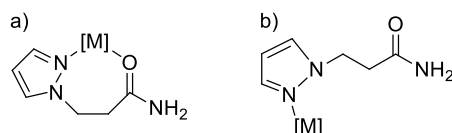


Figure 1. Frequently observed coordination patterns in transition metal complexes with 3-(pyrazol-1-yl)propanamide (PPA) ligands: $\kappa\text{N},\kappa\text{O}$ -chelating (a), and κN -monodentate (b).

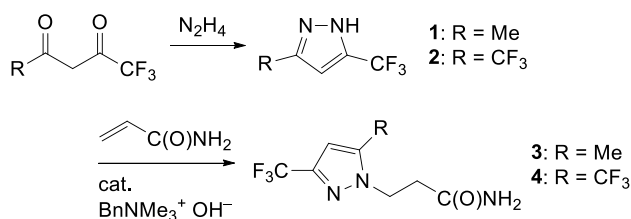
PPA ligands are deliberately available in one step by Aza-Michael addition of the specific pyrazole derivative to acrylamide.^[14,15,17–19] This allows for the facile introduction of substituents at the ethylene backbone,^[17] the pyrazolyl group,^[17,18,20] or the amide nitrogen atom.^[19] While these modifications influence predominantly the steric properties and/or the hydrogen-bonding ability of the ligand, the electronic properties of all previously reported PPA derivatives did not differ very much. In the

present study, we were interested in PPA ligands having trifluoromethyl as a strongly electron-withdrawing substituent at the pyrazolyl group, potentially resulting in a significantly different complex formation behavior of the ligand. We report here the synthesis of two new PPA ligands, their extensive analytical and spectroscopic characterization, and their reactions with palladium(II) chloride. The properties and structures of the resulting complexes are discussed.

Results and Discussion

The starting materials 3-methyl-5-trifluoromethylpyrazole (**1**)^[21] and 3,5-bis(trifluoromethyl)pyrazole (**2**)^[22] were obtained by a standard method, treating the corresponding 1,3-diketones with hydrazine (Scheme 1). Subsequent treatment of **1** and **2** with acrylamide following a standard protocol^[14] afforded the new 3-pyrazolylpropanamides CF_3MePPA (**3**) and $(\text{CF}_3)_2\text{PPA}$ (**4**). A great advantage of the trifluoromethylated pyrazoles is that these reactions can be easily monitored by ^{19}F NMR spectroscopy. Thereby, it was found that **1** showed a reactivity toward acrylamide comparable to the unsubstituted pyrazole, and the reaction proceeded to completion after 6 h in refluxing methanol. The nucleophilic addition of the pyrazole is highly regioselective, and only one out of two different isomers of **3** was obtained. This was identified as the 3-trifluoromethyl-5-methyl isomer by X-ray structure determination (*vide infra*). Compound **2** appeared to be less reactive than **1** and about 80% NMR yield of **4** was reached after 28 h of reflux using an excess of acrylamide. The new PPA derivatives were isolated in good yields by recrystallization from water. Both products melt without decomposition around 100 °C. Both **3** and **4** were characterized by the usual set of analytical techniques, including elemental analyses (C, H, N), ATR-IR and NMR (^1H , ^{13}C , ^{19}F) spectroscopy, and mass spectrometry. While **4** was obtained as extremely fine needles that resisted all attempts for single-crystal X-ray structure determination, the crystal quality of **3** allowed for elucidation of the solid-state structure. The asymmetric unit contains one distinct CF_3MePPA

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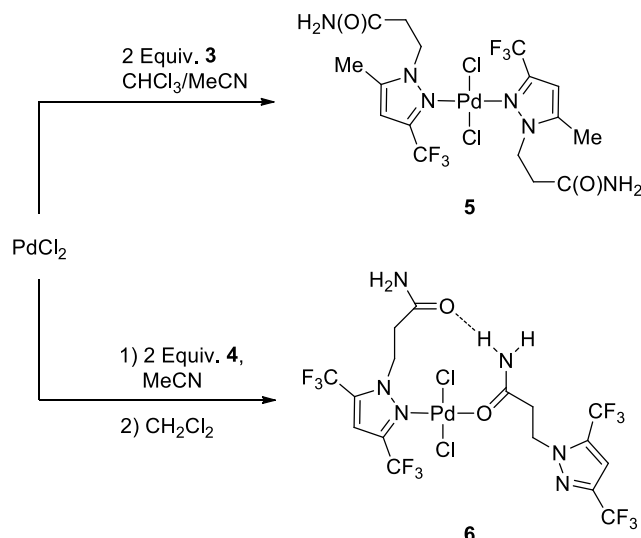
Scheme 1. Synthetic route to the new 3-(pyrazol-1-yl)propanamide (PPA) derivatives **3** and **4**.

molecule with the trifluoromethyl and methyl groups in 3- and 5-position of the pyrazole ring, respectively (Figure 2, left). As it has been previously seen with related PPA derivatives,^[14,17,18,20] the molecules are linked into an infinite assembly by hydrogen bonds. Thereby, a similar picture as for 3-MePPA^[17] and 3-BuPPA^[18] was observed, thus forming a one-dimensional, ribbon-like polymeric structure by N-H...O and N-H...N(pyrazolyl) bonds (*cf.* Figure S2 in the Supplementary Material), while for the unsubstituted PPA^[14] and Me₂PPA^[20] two-dimensional supramolecular structures have been observed.

In order to investigate the ligating properties of the new PPA derivatives **3** and **4**, we studied their reactions with PdCl₂. The palladium salt dissolved in a boiling chloroform solution of **3**, resulting in the deliberate formation of PdCl₂(CF₃MePPA)₂ (**5**; Scheme 2). This reaction could be accelerated by addition of acetonitrile to the solution, increasing the solubility of the palladium salt. Complex **5** was observed as a sparingly soluble, yellow powder, which decomposes thermally not below 200 °C and could be isolated in 78% yield after removal of minor amounts of elemental Pd by Soxhlet extraction. The preparation of defined complexes with the bis-trifluoromethylated ligand **4** turned out to be more challenging. No reaction was observed under comparable conditions as for **3**, and in more polar solvents such as methanol, the only isolable product was PdCl₂(H₂O)₂ besides free **4** and elemental palladium due to decomposition. However, the result was different when the reaction was run in dry acetonitrile under exclusion of air. Under these conditions, PdCl₂(MeCN)₂^[23] is initially formed, which could be transformed into PdCl₂((CF₃)₂PPA)₂ (**6**) by subsequent change of the solvent to CH₂Cl₂. This product could be isolated in moderate yield by fractionated crystallization and is thermally stable up to 116 °C. At least one other (CF₃)₂PPA complex was detected in the product mixture (tentatively a mixed-ligand complex containing both (CF₃)₂PPA and MeCN), which was inseparable from PdCl₂(MeCN)₂.

The IR spectrum of complex **5** in the solid state shows one strong ν(C=O) band at 1685 cm⁻¹, which is slightly shifted to smaller wavenumbers as compared to the free ligand **3** (1694 cm⁻¹). This could be due to the presence of stronger N-H...O bonds in **5**, while metal coordination of the C=O group can be ruled out (see the crystal structure of **5**; *vide infra*). The IR spectrum of **6** displays two bands in the carbonyl region (1695 and 1638 cm⁻¹), which are shifted to higher energies as compared to the free ligand **4** (1675 and 1622 cm⁻¹). This might partially be attributed to the fact that the Pd-O coordination in **6** is weaker than an N-H...O bond and does therefore impact the C=O bond to a lesser extent (see the crystal structure of **6**; *vide infra*). The carbonyl band positions for **7** are 1674 cm⁻¹ and 1624

cm⁻¹, and therefore more similar to free **4** than to complex **6**. This suggests that the amide group in **7** is involved in N-H...O bonding rather than κO-coordination, and the PPA ligand is in a typical κN-monodentate mode.



Scheme 2. Preparation of PdCl₂ complexes with the new PPA ligands **3** and **4**.

For both complexes **5** and **6**, the NMR spectra displayed one set of signals for the respective PPA ligand. For **6**, which contains two differently bonded (CF₃)₂PPA ligands in the solid state (*vide infra*), this could be due to fast dynamic exchange of the relatively weakly bonded ligands. In the ¹H NMR spectrum of **5**, the signals are significantly downfield-shifted as compared to the free ligand **3** (ca. 0.9 ppm for both CH₂ signals and ca. 0.3 ppm for 4-CH; Table 1). This behavior is very similar to what has been seen with Me₂PPA and its PdCl₂ complex.^[18,19] By contrast, the ¹H NMR signals of the (CF₃)₂PPA ligand (**4**) show virtually no coordination shift in **6**, corroborating the assumption that **4** is a considerably weaker ligand than **3** and the specific complexes might dissociate readily in CD₂Cl₂ solution. This picture was confirmed by the ¹⁹F NMR spectra, containing a signal for **5** which is 2.7 ppm downfield-shifted as compared to **3**, while the chemical shift values observed for **4** and **6** were virtually identical (see the Supplementary Material for details). Due to solubility issues, comparison of the ¹³C NMR shifts with those of the free ligand was only possible for **6**. Thereby, again very similar values as for **4** were obtained.

X-ray structure determination of **5** revealed the presence of centrosymmetric PdCl₂(CF₃MePPA)₂ molecules, in which two *trans*-arranged CF₃MePPA ligands are attached to the Pd atom in a κN-monodentate mode (Figure 2, middle). Complex **5** is structurally related to previously reported palladium complexes such as PdCl₂(Me₂PPA)₂.^[19] The Pd-N bond lengths are 201.5(2) pm and therefore only marginally longer than in previously reported PdCl₂(PPA)₂-type complexes (200.2(2)–200.9(1) pm). This finding shows that the influence of the trifluoromethyl group in 3-position of the pyrazolyl ring on the ligating properties of the PPA ligand is weak. However, the hydrogen bonding pattern in the crystal structure of **5** is different than for related complexes,^[19] showing *intermolecular* N-H...Cl bonds instead of the usually observed *intramolecular* N-H...Cl bonds (*cf.* Figure S4). The remaining N-H donor is, similar as for the known Me₂PPA

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Table 1. Selected ^1H and ^{13}C NMR shifts (ppm) for compounds **3–6**, and related compounds. NMR spectra were recorded in CD_2Cl_2 solution at ambient temperature.

Compound	$\delta_{\text{H}}(\alpha\text{-CH}_2)$	$\delta_{\text{H}}(\beta\text{-CH}_2)$	$\delta_{\text{H}}(4\text{-CH})$	$\delta_{\text{C}}(\text{CONH}_2)$	$\delta_{\text{C}}(\alpha\text{-CH}_2)$	$\delta_{\text{C}}(\beta\text{-CH}_2)$	$\delta_{\text{C}}(3\text{-C})$	$\delta_{\text{C}}(4\text{-CH})$	$\delta_{\text{C}}(5\text{-C})$
$\text{Me}_2\text{PPA}^{[\text{a}]}$ [18]	2.54	4.06	5.73	171.7	35.5	43.9	145.8	104.3	138.6
$\text{PdCl}_2(\text{Me}_2\text{PPA})_2^{[\text{b},\text{c}]}$ [19]	3.79	4.98	5.91						
CF_3MePPA (3)	2.83	4.32	6.25	172.6	35.6	45.4	141.6	103.9	141.5
$\text{PdCl}_2(\text{CF}_3\text{MePPA})_2^{[\text{c}]}$ (5)	3.72	5.20	6.51						
$(\text{CF}_3)_2\text{PPA}$ (4)	2.91	4.56	6.91	171.8	35.0	48.0	142.1	106.7	134.0
$\text{PdCl}_2\{(\text{CF}_3)_2\text{PPA}\}_2$ (6)	2.93	4.58	6.92	171.4	34.9	48.0	142.1	106.8	134.0

^[a] measured in $\text{DMSO}-d_6$. ^[b] measured in CDCl_3 . ^[c] due to low solubility, no satisfying ^{13}C NMR spectrum was obtained.

complex, involved in intermolecular $\text{N-H}\cdots\text{O}$ bonding between the non-coordinated amide moieties. The combination of $\text{N-H}\cdots\text{Cl}$ and $\text{N-H}\cdots\text{O}$ linkages in **5** leads to the formation of a close two-dimensional array, which might be the origin of the low solubility of this compound.

The solid-state structure of **6** comprises non-centrosymmetric molecules, in which only one of the two $(\text{CF}_3)_2\text{PPA}$ ligands adopts the usual κN -monodentate coordination (Figure 2, right). The second ligand is bond to the Pd atom in a κO -monodentate mode and additionally fixed by an intramolecular $\text{N-H}\cdots\text{O}$ bond between the N-H of the coordinated amide group and the carbonyl group of the non-coordinated amide group. The Pd-N bond length is 200.0(5) pm and therefore slightly shorter than in **5**, which could be due to the weaker *trans* influence of the amide group as compared to another pyrazolyl unit. The Pd-O bond is 201.2(5) pm and resembles the values observed for other Pd complexes with κO -monodentate amide ligands, which are relatively rare in the literature. Examples are *trans*- $\text{PdCl}_2(\text{N,N}$ -dimethylacetamide) $_2$ (Pd-O 200.4(1) pm) $^{[24]}$ and *trans*- $\text{Pd}(\text{NO}_3)_2(\text{N}$ -cyclohexylpyrrolidine-2-one) $_2$ (Pd-O 200.9(1) and 201.1(2) pm). $^{[25]}$ The exposed NH_2 group of the κN - $(\text{CF}_3)_2\text{PPA}$ ligand and the free N-H donor of the κO - $(\text{CF}_3)_2\text{PPA}$ ligand in **6** are involved in intermolecular $\text{N-H}\cdots\text{Cl}$ and $\text{N-H}\cdots\text{O}$ bonding, resulting in a two-stranded, ribbon-like supramolecular structure in the crystal (cf. Figure S6).

Summarizing the results reported here, we have shown that the ligating properties of PPA ligands can be significantly modified by introduction of trifluoromethyl substituents at the pyrazolyl group. While the 3-trifluoromethylated ligand CF_3MePPA (**3**) appeared to exhibit a reactivity toward PdCl_2 similar to Me_2PPA , the bis-trifluoromethylated derivative

$(\text{CF}_3)_2\text{PPA}$ (**4**) was shown to be a considerably weaker ligand. With the latter derivative, we prepared the first complex with an unusual κO -monodentate PPA coordination. As it can be expected that this coordination is relatively weak and the respective PPA ligand can be relatively easily replaced by other ligands, complexes with **4** are interesting for the preparation of mixed-ligand complexes that are potentially useful in homogeneous catalysis. Generally, it seems like substitution in 5-position of the pyrazolyl group seems to exert a stronger influence on the electronic properties of the PPA ligand than substitution in 3-position does. This postulate could be corroborated by investigation of the respective ligand with a CF_3 substituent exclusively in 5-position. Moreover, in future research it would be interesting to introduce other substituents in 5-position, and to evaluate the influence of substituents in 4-position of the pyrazolyl group.

Supplementary Material

Supporting information for this article is available on the WWW under <http://dx.doi.org/10.1002/MS-number>. CCDC 2016132 (**3**), 2016133 (**5**), and 2016134 (**6**) contain the crystallographic data for this paper and are provided free of charge at www.ccdc.cam.ac.uk/structures.

Acknowledgements

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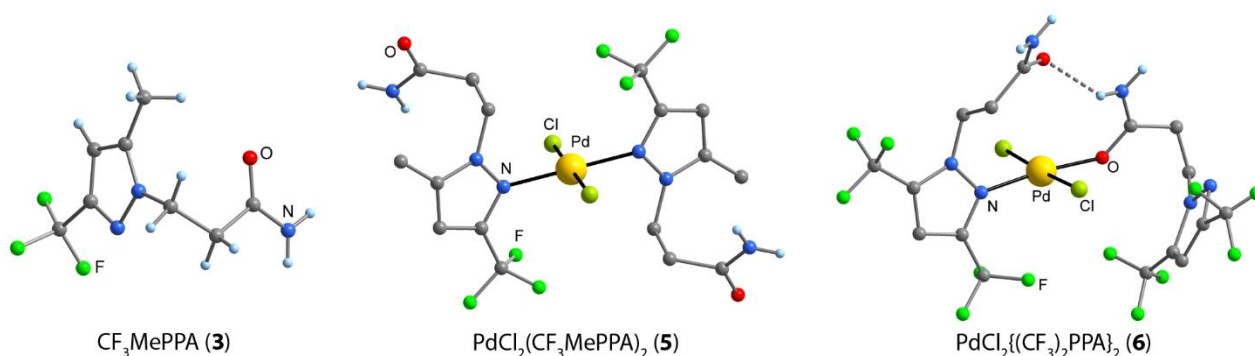


Figure 2. Molecular structures of compounds **3**, **5**, and **7** in the solid state.

Author Contribution Statement

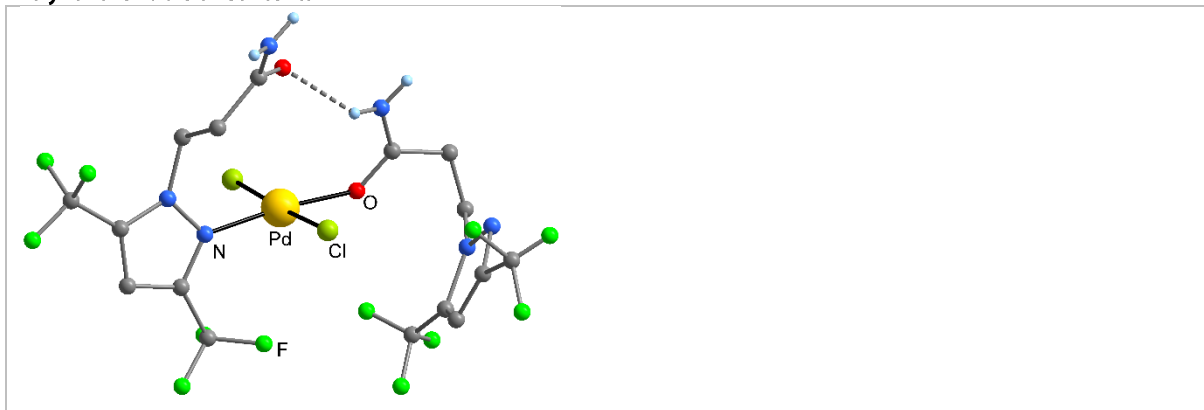
P.L. conceived the project and performed the experimental work. F.T.E. supervised the project. The manuscript was written and edited by P.L. and F.T.E.

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Entry for the Table of Contents



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Trifluoromethylation of 3-(pyrazol-1-yl)-propanamide (PPA) ligands enables the formation of unusual palladium complexes having a κO -monodentate PPA ligand.