

# Synthesis and Structural Characterization of the Gold Complexes of 1,2,4-Triazole Derived N-Heterocyclic Carbene Ligands

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**Abstract** Two gold(I) complexes of an amido-N functionalized 1,2,4-triazole derived N-heterocyclic carbene ligands have been synthesized and structurally characterized. In particular, the {[1-(R)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazol-5-ylidene]<sub>2</sub>Au}<sup>+</sup>Cl<sup>-</sup> [R = Et (**1c**) and *i*-Pr (**2c**)] complexes were conveniently synthesized from their respective silver(I) analogs (**1–2b**) by the direct reaction with (SMe<sub>2</sub>)AuCl in CH<sub>3</sub>CN at room temperature. The molecular structure determination of the gold(I) (**1–2c**) complexes using single crystal X-ray diffraction study revealed a *syn*-disposition of two N-heterocyclic carbene (NHC) ligands bound across the Au(I) center in a linear fashion with its two amido-N functionalized sidearms interacting with a non-coordination Cl anion through two intramolecular H-bonds.

**Keywords** Gold · Silver · N-heterocyclic carbenes · Synthesis · Molecular structure

## 1 Introduction

Of late, the gold complexes have attracted attention particularly for their resurgence in catalytic applications alongside of their longstanding utility in materials and in

biomedical applications research [1–3]. This has led to an unprecedented development of the gold chemistry in recent years beginning with their synthesis and leading to various application studies [4–7]. In this connection, we became interested in this new found catalytic exploits of gold, long known as an inert coinage metal, and decided to explore its catalytic potential when stabilized over an otherwise catalytically renowned ligand like the N-heterocyclic carbenes (NHCs) [8]. Towards this objective, we have reported the utility of the gold N-heterocyclic carbene complexes in the alkyne hydroamination reaction [9, 10], in the Ring-Opening Polymerization (ROP) of L-lactides [11, 12] and in the synthesis of β-enaminones from 1,3-dicarbonyl compounds and aliphatic amines [13]. In addition, we have observed an interesting and also unprecedented long-range 1,7-bromination reaction occurring in these complexes [14], the structural diversity [15] and their antimicrobial properties [16].

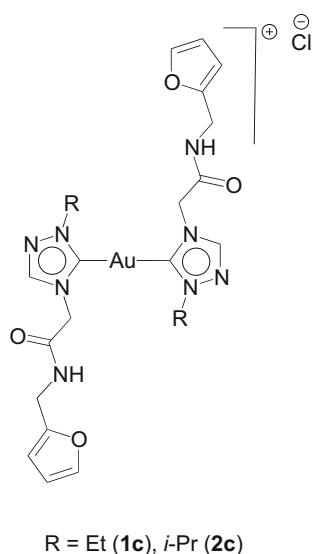
The other emphasis of our recent research is on broadening the horizons of the triazole derived N-heterocyclic carbene ligands [17], which thus far have remained surprisingly overlooked in comparison to their imidazole counterparts. Towards this objective, we have recently noted their utility in borylation of aryl bromides [18], and in hydroaminations of alkynes [10] and activated olefins [19]. Continuing further along the line, we decided to synthesize gold complexes of the triazole derived N-heterocyclic carbene ligands for their potential catalytic applications.

Here in this manuscript, we report the synthesis and the structural characterization of two such gold(I) complexes stabilized over 1,2,4-triazole derived N-heterocyclic carbene ligands containing an amido-N functionalized sidearm substituent (Fig. 1).

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**Fig. 1** Au–NHC complexes of 1,2,4-triazole derived N-heterocyclic carbene ligands

## 2 Experimental Section

### 2.1 Materials and Methods

All manipulations were carried out using a combination of a glovebox and standard Schlenk techniques. 1-Ethyl-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazolium chloride (**1a**), and 1-(*i*-propyl)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazolium chloride (**2a**) were synthesized according to modified literature procedures [18].  $^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  NMR spectra were recorded on a Bruker 400 MHz and Bruker 500 MHz NMR spectrometer.  $^1\text{H}$  NMR peaks are labeled as singlet (s), doublet (d), triplet (t), quartet (q), quartet of doublets (qd), doublet of doublets (dd) septet (sept) and broad (br). Infrared spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrometer. Mass spectrometry measurements were done on a Micromass Q-ToF spectrometer and Bruker maxis impact spectrometer. Elemental Analysis was carried out on Thermo Finnigan FLASH EA 1112 SERIES (CHNS) Elemental Analyzer. X-ray diffraction data for compounds **1c** and **2c** were collected on an Oxford Diffraction XCALIBUR-S diffractometer and Rigaku Hg 724 + diffractometer. Crystal data collection and refinement parameters are summarized in Table 1. The structures were solved using direct method and standard difference map techniques, and refined by full-matrix least-squares procedures on  $F^2$  [21, 22]. CCDC-990570 (for **1c**) and CCDC-814958 (for **2c**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data center via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

**Table 1** X-ray crystallographic data for (**1–2**)c

Compound	<b>1c</b>	<b>2c</b>
Lattice	Triclinic	Monoclinic
Formula	$\text{C}_{46}\text{H}_{59}\text{Au}_2\text{Cl}_2\text{N}_{17}\text{O}_8$	$\text{C}_{24}\text{H}_{32}\text{AuClN}_8\text{O}_4$
Formula weight	1442.94	728.99
Space group	<i>P</i> 1	<i>P</i> 2 <sub>1</sub> / <i>c</i>
<i>a</i> /Å	8.199	14.1457 (3)
<i>b</i> /Å	12.171	10.0426 (2)
<i>c</i> /Å	28.756	21.6925 (4)
$\alpha$ /°	88.72	90
$\beta$ /°	84.41	106.192 (2)
$\gamma$ /°	76.04	90
<i>V</i> /Å <sup>3</sup>	2771.4	2959.39 (10)
<i>Z</i>	2	4
Temperature (K)	100 (2)	150 (2)
Radiation ( $\lambda$ , Å)	0.71073	0.71073
$\rho$ (calcd.), g cm <sup>-3</sup>	1.729	1.636
$\theta$ max, deg.	25.50	25.00
No. of data	14,988	5193
No. of parameters	677	384
<i>R</i> <sub>1</sub>	0.0686	0.0255
<i>wR</i> <sub>2</sub>	0.1092	0.0688
GOF	0.715	1.046

### 2.2 Synthesis of {[1-(ethyl)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazol-5-ylidene]<sub>2</sub>Ag}<sup>+</sup>Cl<sup>-</sup> (**1b**)

A mixture of 1-ethyl-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazolium chloride (**1a**) (0.728 g, 2.68 mmol) and Ag<sub>2</sub>O (0.313 g, 1.34 mmol) was stirred in CH<sub>3</sub>CN (ca. 40 ml) at room temperature for 12 h. The reaction mixture was filtered over Celite and the filtrate was finally dried under vacuum to give the product **1b** as a brown hygroscopic solid (0.585 g, 70 %)  $^1\text{H}$  NMR (CDCl<sub>3</sub>, 500 MHz, 25 °C):  $\delta$  9.00 (br, 2H, NHCO), 8.37 (s, 2H, N–C(3)H–N), 7.20 (br, 2H, C<sub>4</sub>H<sub>3</sub>O), 6.21 (br, 2H, C<sub>4</sub>H<sub>3</sub>O), 6.19 (t, 2H,  $^3J_{\text{HH}} = 2$  Hz, C<sub>4</sub>H<sub>3</sub>O), 5.24 (s, 4H, CH<sub>2</sub>), 4.38 (d, 4H,  $^3J_{\text{HH}} = 5$  Hz, CH<sub>2</sub>NH), 4.33 (q, 4H,  $^3J_{\text{HH}} = 7$  Hz, CH<sub>2</sub>CH<sub>3</sub>), 1.51 (t, 6H,  $^3J_{\text{HH}} = 7$  Hz, CH<sub>2</sub>CH<sub>3</sub>).  $^{13}\text{C}\{^1\text{H}\}$  NMR (DMSO-*d*<sub>6</sub>, 100 MHz, 25 °C):  $\delta$  181.2 (Ag–NCN), 165.9 (C = O), 151.5 (C<sub>4</sub>H<sub>3</sub>O), 144.9 (N–C(3)–N), 142.3 (C<sub>4</sub>H<sub>3</sub>O), 110.5 (C<sub>4</sub>H<sub>3</sub>O), 107.3 (C<sub>4</sub>H<sub>3</sub>O), 50.1 (CH<sub>2</sub>), 48.0 (CH<sub>2</sub>CH<sub>3</sub>), 35.8 (CH<sub>2</sub>), 15.7 (CH<sub>2</sub>CH<sub>3</sub>). IR data (KBr pellet) cm<sup>-1</sup>: 1690 (s) ( $\nu_{\text{C=O}}$ ). HRMS (ES): *m/z* 575.1299 [(NHC)<sub>2</sub> + Ag]<sup>+</sup>, calcd. 575.1284. Anal. Calcd. for C<sub>22</sub>H<sub>28</sub>AgClN<sub>8</sub>O<sub>4</sub>·2H<sub>2</sub>O: C, 40.79; H, 4.98; N, 17.30. Found: C, 40.14; H, 4.92; N, 16.89 %.

### 2.3 Synthesis of {[1-(ethyl)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazol-5-ylidene]<sub>2</sub>Au}<sup>+</sup>Cl<sup>-</sup> (**1c**)

A mixture of {[1-(ethyl)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazol-5-ylidene]<sub>2</sub>Ag}<sup>+</sup>Cl<sup>-</sup> (**1b**) (0.521 g, 0.853 mmol) and (SMe<sub>2</sub>)AuCl (0.250 g, 0.850 mmol) was stirred in CH<sub>3</sub>CN (ca. 40 ml) at room temperature for 12 h, during which the formation of an off-white precipitate of AgCl was observed. The reaction mixture was filtered and filtrate was concentrated under vacuum to give the crude product as a yellow solid. The crude product was finally purified by column chromatography using silica gel as a stationary phase by elution with a mixed medium of CHCl<sub>3</sub>/MeOH (94: 6 v/v) to give the product **1c** as a white solid (0.501 g, 84 %). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz, 25 °C): δ 9.75 (br, 2H, NHCO), 8.44 (s, 2H, N-C(3)H-N), 7.11 (br, 2H, C<sub>4</sub>H<sub>3</sub>O), 6.21 (br, 2H, C<sub>4</sub>H<sub>3</sub>O), 6.14 (br, 2H, C<sub>4</sub>H<sub>3</sub>O), 5.27 (s, 4H, CH<sub>2</sub>), 4.38–4.35 (m, 8H, CH<sub>2</sub>CH<sub>3</sub> and CH<sub>2</sub>-NH), 1.56 (t, 6H, <sup>3</sup>J<sub>HH</sub> = 7 Hz, CH<sub>2</sub>CH<sub>3</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ 184.8 (Au-NCN), 165.7 (C=O), 151.5 (C<sub>4</sub>H<sub>3</sub>O), 144.5 (N-C(3)-N), 141.9 (C<sub>4</sub>H<sub>3</sub>O), 110.4 (C<sub>4</sub>H<sub>3</sub>O), 107.8 (C<sub>4</sub>H<sub>3</sub>O), 50.8 (CH<sub>2</sub>), 48.9 (CH<sub>2</sub>CH<sub>3</sub>), 36.5 (CH<sub>2</sub>), 15.7 (CH<sub>2</sub>CH<sub>3</sub>). IR data (KBr pellet) cm<sup>-1</sup>: 1685 (s) (ν<sub>C=O</sub>). HRMS (ES): *m/z* 665.1907 [(NHC)<sub>2</sub> + Au]<sup>+</sup>, calcd. 665.1894. Anal. Calcd. for C<sub>22</sub>H<sub>28</sub>AuClN<sub>8</sub>O<sub>4</sub>: C, 37.70; H, 4.03; N, 15.99. Found: C, 37.55; H, 4.77; N, 16.69 %.

### 2.4 Synthesis of {[1-(*i*-propyl)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazol-5-ylidene]<sub>2</sub>Ag}<sup>+</sup>Cl<sup>-</sup> (**2b**)

A mixture of 1-(*i*-propyl)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazolium chloride (**2a**) (1.95 g, 6.84 mmol) and Ag<sub>2</sub>O (0.799 g, 3.45 mmol) was stirred in CH<sub>3</sub>CN (ca. 40 mL) at room temperature for 12 h. The reaction mixture was filtered over Celite and the filtrate was finally dried under vacuum to give the product **2b** as a brown hygroscopic solid (0.999 g, 45 %). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ 9.05 (br, 2H, NHCO), 8.37 (s, 2H, N-C(3)H-N), 7.20 (br, 2H, C<sub>4</sub>H<sub>3</sub>O), 6.22 (br, 2H, C<sub>4</sub>H<sub>3</sub>O), 6.20–6.19 (m, 2H, C<sub>4</sub>H<sub>3</sub>O), 5.23 (s, 4H, CH<sub>2</sub>), 4.82 (sept, 2H, <sup>3</sup>J<sub>HH</sub> = 7 Hz, CH(CH<sub>3</sub>)<sub>2</sub>), 4.37 (d, 4H, <sup>3</sup>J<sub>HH</sub> = 5 Hz, CH<sub>2</sub>NH), 1.52 (d, 6H, <sup>3</sup>J<sub>HH</sub> = 7 Hz, CH(CH<sub>3</sub>)<sub>2</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (DMSO-*d*<sub>6</sub>, 100 MHz, 25 °C): δ 180.9 (Ag-NCN), 166.1 (C=O), 151.5 (C<sub>4</sub>H<sub>3</sub>O), 144.7 (N-C(3)-N), 142.3 (C<sub>4</sub>H<sub>3</sub>O), 110.5 (C<sub>4</sub>H<sub>3</sub>O), 107.3 (C<sub>4</sub>H<sub>3</sub>O), 55.2 (CH(CH<sub>3</sub>)<sub>2</sub>), 50.2 (CH<sub>2</sub>), 35.7 (CH<sub>2</sub>), 21.8 (CH(CH<sub>3</sub>)<sub>2</sub>). IR data (KBr pellet) cm<sup>-1</sup>: 1685 (s) (ν<sub>C=O</sub>). HRMS (ES): *m/z* 603.1584 [(NHC)<sub>2</sub> + Ag]<sup>+</sup>, calcd. 603.1597. Anal. Calcd. for C<sub>24</sub>H<sub>32</sub>AgClN<sub>8</sub>O<sub>4</sub>·3H<sub>2</sub>O: C, 41.54; H, 5.52; N, 16.15. Found: C, 40.95; H, 4.56; N, 15.21 %.

### 2.5 Synthesis of {[1-(*i*-propyl)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazol-5-ylidene]<sub>2</sub>Au}<sup>+</sup>Cl<sup>-</sup> (**2c**)

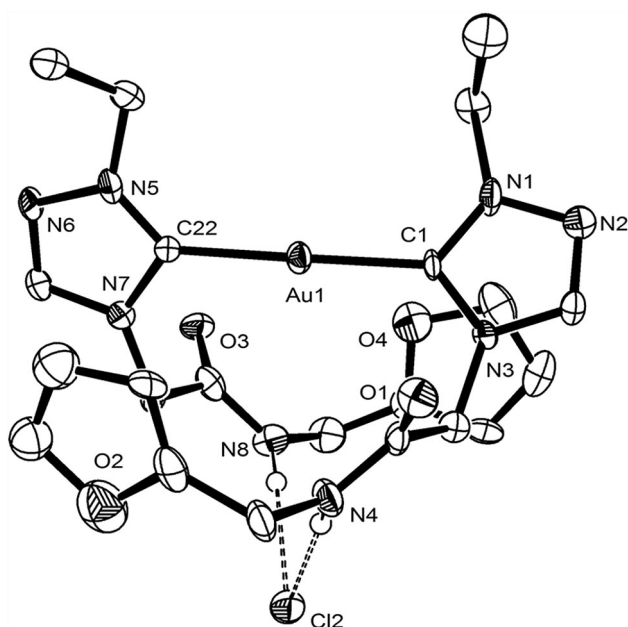
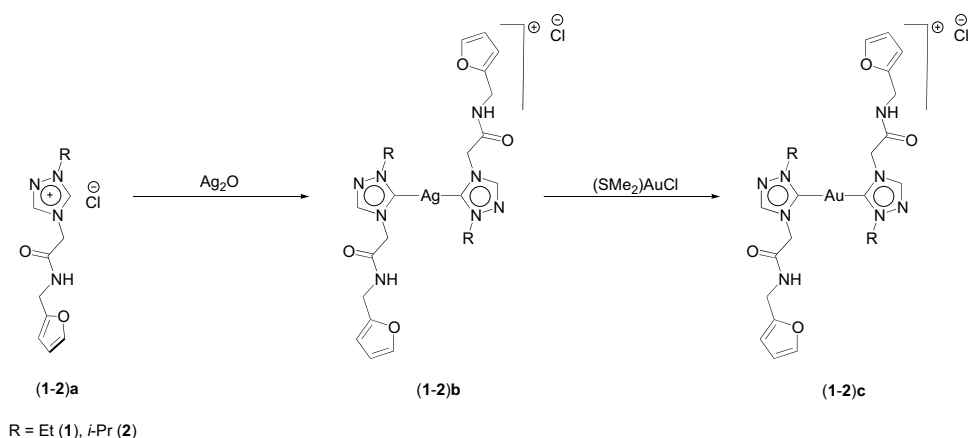
A mixture of {[1-(*i*-propyl)-4-N-(furan-2-ylmethyl)acetamido-1,2,4-triazol-5-ylidene]<sub>2</sub>Ag}<sup>+</sup>Cl<sup>-</sup> (**2b**) (0.778 g, 1.21 mmol) and (SMe<sub>2</sub>)AuCl (0.178 g, 0.606 mmol) was stirred in CH<sub>3</sub>CN (ca. 40 ml) at room temperature for 12 h, during which the formation of an off-white precipitate of AgCl was observed. The reaction mixture was filtered and filtrate was concentrated under vacuum to give the crude product as a yellow solid. The crude product was finally purified by column chromatography using silica gel as a stationary phase and elution with a mixed medium of CHCl<sub>3</sub>/MeOH (94: 6 v/v) to give the product **2c** as a white solid (0.145 g, 34 %). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ 9.88 (br, 1H, NHCO), 8.39 (s, 1H, N-C(3)H-N), 7.13 (d, 1H, <sup>3</sup>J<sub>HH</sub> = 2 Hz, C<sub>4</sub>H<sub>3</sub>O), 6.32 (dd, 1H, <sup>3</sup>J<sub>HH</sub> = 3 Hz, C<sub>4</sub>H<sub>3</sub>O), 6.25 (dd, 1H, <sup>3</sup>J<sub>HH</sub> = 3 Hz, C<sub>4</sub>H<sub>3</sub>O), 5.28 (s, 2H, CH<sub>2</sub>), 4.90 (sept, 1H, <sup>3</sup>J<sub>HH</sub> = 7 Hz, CH(CH<sub>3</sub>)<sub>2</sub>), 4.42 (d, 2H, <sup>3</sup>J<sub>HH</sub> = 6 Hz, CH<sub>2</sub>NH), 1.60 (d, 6H, <sup>3</sup>J<sub>HH</sub> = 7 Hz, CH(CH<sub>3</sub>)<sub>2</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C) δ 184.0 (Au-NCN), 165.7 (C=O), 151.7 (C<sub>4</sub>H<sub>3</sub>O), 144.3 (N-C(3)-N), 141.9 (C<sub>4</sub>H<sub>3</sub>O), 110.4 (C<sub>4</sub>H<sub>3</sub>O), 107.8 (C<sub>4</sub>H<sub>3</sub>O), 56.4 (CH(CH<sub>3</sub>)<sub>2</sub>), 51.0 (CH<sub>2</sub>), 36.5 (CH<sub>2</sub>), 22.9 (CH(CH<sub>3</sub>)<sub>2</sub>). IR data (KBr pellet) cm<sup>-1</sup>: 1686 (s) (ν<sub>C=O</sub>). Anal. Calcd. for C<sub>24</sub>H<sub>32</sub>AuClN<sub>8</sub>O<sub>4</sub>·CH<sub>3</sub>CN: C, 40.55; H, 4.58; N, 16.37. Found: C, 40.46; H, 4.98; N, 17.02 %.

## 3 Results and Discussion

The gold(I) (**1–2**)c complexes were synthesized from the respective silver(I) analogs (**1–2**)b by the reaction with (SMe<sub>2</sub>)AuCl in CH<sub>3</sub>CN at room temperature (Scheme 1) and the workup required product purification by column chromatography. The silver(I) (**1–2**)b complexes were obtained by the treatment of the corresponding triazolium chloride salts (**1–2**)a [18] with Ag<sub>2</sub>O in moderate to good yields. It is noteworthy that despite repeated attempts the single crystals of the silver(I) (**1–2**)b complexes could not be obtained for structural characterization studies.

The molecular structure of the gold(I) (**1–2**)c complexes, as determined by the single crystal X-ray diffraction study, revealed discrete monomeric nature of these complexes (Figs. 2, 3). The Au(I) center was coordinated to the N-heterocyclic carbene ligand in a linear fashion in concurrence with the *d*<sup>10</sup> configuration of the central metal ion. The two N-heterocyclic carbene ligands are bound *trans* to each other displaying a *syn*-disposition of its substituents, with its amido-N substituent engaging with the non-coordinating Cl anion with the aid of two intramolecular H-bonding interactions. Similar *syn*-

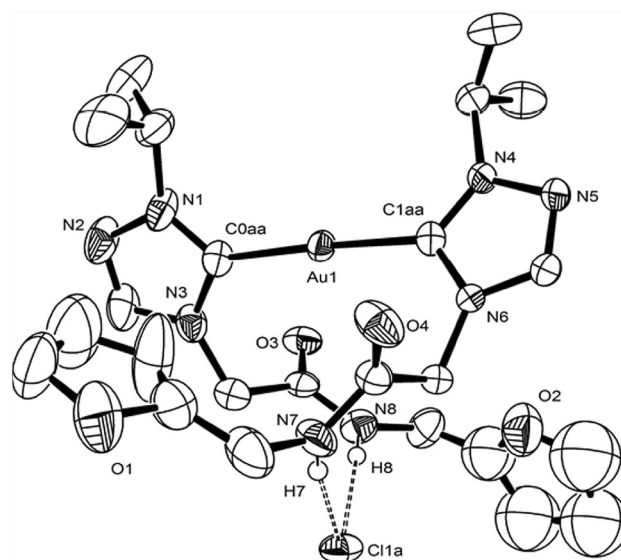
**Scheme 1** Synthetic procedure for the Au–NHC complexes (1–2)c



**Fig. 2** ORTEP of **1c** with thermal ellipsoids are shown at the 50 % probability level. Selected bond lengths (Å) and angles (°): Au1–C1 2.017(7), Au1–C22 2.025(7), C22–Au1–C1 177.2(3), N1–C1–N3 103.4(6), N5–C22–N7 104.4(6)

disposition of the amido-N substituents arising from the intramolecular H-bonding interactions of the amido-NH moieties with the non-coordinating Cl<sup>−</sup> anion has been observed earlier in the silver(I) and gold(I) analogs, {[1-(R)-3-{*N*-(*t*-butylacetamido)imidazol-2-ylidene}]<sub>2</sub>M}<sup>+</sup>Cl<sup>−</sup> (M = Ag, Au; R = *i*-Pr, *t*-Bu), of the related imidazole based N-heterocyclic carbene ligands [15]. Quite interestingly, no intermolecular H-bonding interaction was present among these discrete monomeric molecules in the crystal lattice.

Of particular interest are the Au–C<sub>carbene</sub> bond distances in the **1c** [2.017(7) Å, 2.025(7) Å] and the **2c** [2.026(6) Å,



**Fig. 3** ORTEP of **2c** with thermal ellipsoids are shown at the 50 % probability level. Selected bond lengths (Å) and angles (°): Au1–C1aa 2.026(6), Au1–C0aa 2.014(6), C1aa–Au1–C0aa 177.0(2), N1–C0aa–N3 103.2(5), N4–C1aa–N6 103.8(5)

2.014(6) Å] complexes, that are marginally shorter than the sum of the individual covalent radii of Au and C (2.09 Å) [20] but compare well with that of the representative related imidazole analogs, {[1-(*i*-propyl)-3-{*N*-(*t*-butylacetamido)imidazol-2-ylidene}]<sub>2</sub>Au}<sup>+</sup>Cl<sup>−</sup> [2.017(7) Å] [15] and the {[1-(*t*-butyl)-3-{*N*-(*t*-butylacetamido)imidazol-2-ylidene}]<sub>2</sub>Au}<sup>+</sup>Cl<sup>−</sup> [2.028(5) Å] [15]. The angle at the gold center, ∠C<sub>carbene</sub>–Au–C<sub>carbene</sub>, in the **1c** [177.2(3)°] and in the **2c** [177.0(2)°] complexes is close to linearity and is also comparable to that observed for the related representative imidazole analogs, {[1-(*i*-propyl)-3-{*N*-(*t*-butylacetamido)imidazol-2-ylidene}]<sub>2</sub>Au}<sup>+</sup>Cl<sup>−</sup> [178.3(4)°] [15] and the {[1-(*t*-butyl)-3-{*N*-(*t*-butylacetamido)imidazol-2-ylidene}]<sub>2</sub>Au}<sup>+</sup>Cl<sup>−</sup> [179.6(3)°] [15].

## 4 Conclusion

In summary, two gold(I) complexes of the 1,2,4-triazole derived N-heterocyclic carbene complexes (**1–2**)**c** have been synthesized. The single crystal X-ray diffraction study revealed that the (**1–2**)**c** complexes are structurally similar to their related imidazole counterparts known in the literature. Detailed evaluation of these complexes in catalytic applications is currently underway and would be a part of the future publications from the group.

## 5 Supporting Information Available

The  $^1\text{H}$  NMR,  $^{13}\text{C}\{^1\text{H}\}$  NMR, IR, HRMS and the CHN data of the silver (**1–2**)**b** and the gold (**1–2**)**c** complexes; CIF files giving X-ray crystallographic data associated with this article can be found in the journal webpage. This material is available free of charge via the journal webpage.

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