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Synthesis and X-ray crystal structure of a cationic gold (I) π-(1,3-diene) complex generated via isomerization of a gold π-allene complex

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ABSTRACT

The cationic gold tetramethylallene complex {[P(*t*-Bu)$_2$*o*-biphenyl]Au(η^2-Me$_2$C═C═CMe$_2$)}$^+$ SbF$_6^-$ (**1**) underwent formal 1,3-hydrogen migration in CD_2Cl_2 solution at or below room temperature in the absence of base to form the 1,3-diene complex {[P(*t*-Bu)$_2$*o*-biphenyl]Au[η^2-H$_2$C(Me) = C(H)–C(H)═CMe$_2$]}$^+$ SbF$_6^-$ (**2**), which was isolated in 92% yield and analyzed by X-ray crystallography.

1. Introduction

The conversion of an allene to a conjugated diene is a potentially useful transformation owing to the versatility of conjugated dienes as building blocks in organic synthesis. Although the isomerization of allenes activated by heteroatoms can be realized under mild conditions [1,2], effective methods for the conversion of electronically unactivated alkyl and arylallenes to dienes via formal 1,3-hydrogen migration remain scarce despite the large negative enthalpy of isomerization [3]. For example, the thermal isomerization of vinylidenecyclopentane to 1-vinylcyclopent-1-ene occurs with an energy barrier of ~50 kcal/mol [4]. Yamamoto has reported the Pd(0)/acetic acid-catalyzed isomerization of aliphatic 1,1-disubstituted allenes and 1-aryl-1,2-butadienes in modest yield and stereoselectivity [5]. More recently, Liu has reported the $AuCl_3$/nitrosobenzene-catalyzed conversion of arylallenes to 1,3-dienes under mild conditions, but noted that cationic gold(I) complexes were not effective for this transformation [6]. In addition to these catalytic processes, Pettit reported the conversion of the iron tetramethylallene complex (CO)$_4$Fe(π-Me$_2$C═C═CMe$_2$) (**A**) to the iron diene complex (CO)$_3$Fe[η^4-H$_2$C(Me)═C(H)–C(H)═CMe$_2$] upon prolonged heating or though reaction of **A** with Fe$_2$(CO)$_9$ [7].

Extensive research effort over the past decade has established cationic gold(I) complexes as particularly active catalysis for the functionalization of C–C bonds [8]. In an effort to develop a meaningful understanding of the mechanisms of these transformations, there has been considerable recent interest in the synthesis and study of cationic, two-coordinate gold π-complexes that are often invoked as intermediates in these gold(I)-catalyzed transformations [9]. Of these complexes, gold π-allene [10,11] and π-diene [12,13] complexes are particularly intriguing owing to the possibility of multiple coordination modes and fluxional behavior involving the π-ligand. Despite the pronounced π-acidity of a cationic twelve-electron gold fragment (L)Au$^+$, the isomerization of a gold(I) π-allene complex to a π-diene complex via formal 1,3-hydrogen shift has not previously been documented. Here we report isomerization of a cationic gold tetramethylallene complex to the corresponding gold 2,4-dimethyl-2,3-pentadiene complex along with the X-ray crystal structure of the latter complex.

2. Results and discussion

2.1. Conversion of *1* to *2*

We have recently reported isolation of the gold π-tetramethylallene complex [(**P1**)Au(η^2-Me$_2$C═C═CMe$_2$)]$^+$ SbF$_6^-$ [**1**; **P1** = P(*t*-

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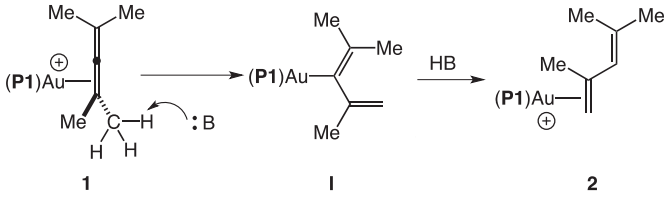
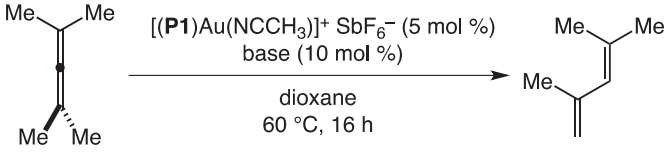
Bu)$_2$*o*-biphenyl] in 96% yield from reaction of tetramethylallene with a 1:1 mixture of (**P1**)AuCl and $AgSbF_6$ in CH_2Cl_2 at room temperature for 15 min [10]. However, attempted crystallization of **1** from a concentrated CH_2Cl_2 solution (75 mM) at 4 °C for 36 h led instead to isomerization via formal 1,3-hydrogen migration to form the gold π-1,3-diene complex {(**P1**)Au[η^2-H_2C=C(Me)–C(H)=CMe_2]}$^+$ SbF_6^- (**2**) (Scheme 1) [12]. Complex **2** was isolated from this reaction as the methylene chloride solvate complex **2**·CH_2Cl_2 in 92% yield as colorless crystals and subsequently analyzed by X-ray crystallography. An enthalpy of reaction for the conversion of **1** to **2** of $\Delta H = -8.6$ kcal/mol was estimated from the calculated heats of formation and binding affinities of 2,4-dimethyl-2,3-pentadiene and 2,4-dimethyl-1,3-pentadiene [14,15].

Precipitation of **2** was not required for isomerization of **1**. For example, when a solution of **1** (55 mM) in CD_2Cl_2 was analyzed periodically by ^{1}H and ^{31}P NMR spectroscopy at room temperature, ~10% of **1** was converted to **2** within 3 h and **1** was completely consumed after 4 days to form **2** in 86% yield (^{1}H NMR). Free tetramethylallene played no important role in the conversion of **1** to **2** as a similar experiment involving a 1:1 solution of **1** and tetramethylallene formed **2** in 77% after 4 days. To probe for a deprotonation event in the conversion of **1** to **2**, a CD_2Cl_2 solution of **1** was treated with triethylamine (1 equiv) at room temperature. ^{1}H and ^{31}P NMR analyses of the resulting solution within 5 min revealed ~50% isomerization and quantitative ligand displacement to form a 2:1:1 mixture of [(**P1**)Au(NEt_3)]$^+$ SbF_6^- (**3**) [16], 2,4-dimethyl-2,3-pentadiene, and 2,4-dimethyl-1,3-pentadiene (Scheme 1). Continued analysis of the reaction mixture revealed slow isomerization of tetramethylallene to form at 1:2 ratio of tetramethylallene and 2,4-dimethyl-1,3-pentadiene after 24 h.

The observations outlined in the proceeding paragraph point to a mechanism for the conversion of **1** to **2** involving deprotonation of the allenyl methyl group of **1** to form the gold σ-dienyl intermediate **I** that undergoes protodeauration to form **2** (Scheme 2). A similar mechanism was proposed on the basis of DFT calculations for the $AuCl_3$/nitrosobenzene-catalyzed isomierzation of 1-arylallenes [17]. In the case of the isomerization of **1** in the presence of triethylamine, deprotonation competes kinetically with irreversible displacement of the tetramethylallene ligand of **1** with the strong σ-donor triethylamine to form **3**. In the absence of exogenous amine, the nature of the base in the conversion of **1** to **2** is unclear. However, we have previously demonstrated the conversion of cationic gold π-(1-arylalkyne) complexes to dinuclear σ,π-acetylide complexes in the absence of base which generated Brønsted acid strong enough to protonate free arylacetylene [18]. In either case, acidification of the allenyl methyl protons of **1** further



Scheme 2.



base: PhN=O (~5%), DBU (<5%), 2,6-di-*t*-Bu-pyridine (<5%), *N*-methylpyrrolidinone (20%), *N*,*N*-dimethylaniline (35%), 2,2,6,6-tetramethylpiperidine (<5%)

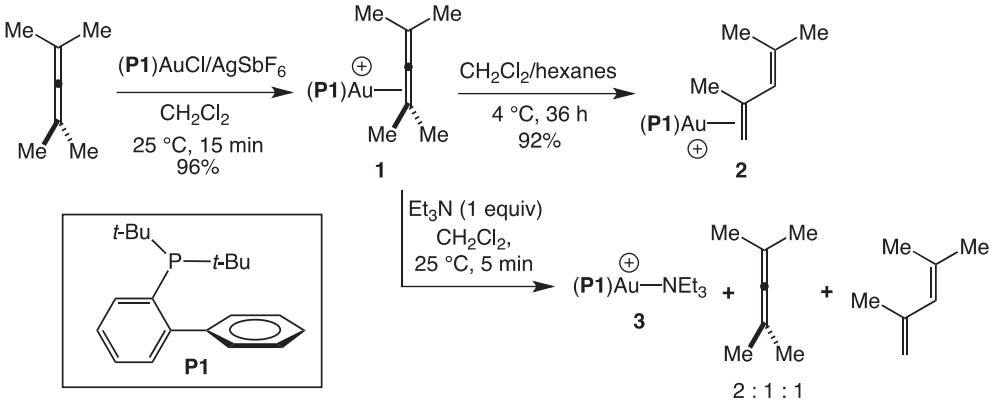
Scheme 3.

establishes the significant π-acidity of the cationic, twelve electron (**P1**)Au$^+$ fragment [15].

Given the rapid, albeit incomplete, conversion of the 2,4-dimethyl-2,3-pentadiene ligand of **1** to free 2,4-dimethyl-1,3-pentadiene in the presence of triethylamine, we considered that cationic gold(I) complexes might catalyze the conversion of 2,4-dimethyl-2,3-pentadiene to 2,4-dimethyl-1,3-pentadiene in the presence of a weakly coordinating base. To this end, a dioxane solution of 2,4-dimethyl-2,3-pentadiene (0.1 M), [(**P1**)Au($NCCH_3$)]$^+$ SbF_6^- (5 mol %), and an exogenous base (10 mol %) was heated at 60 °C for 16 h (Scheme 3). Of the bases tested, *N*,*N*-dimethylaniline proved most effective, providing 2,4-dimethyl-1,3-pentadiene in 35% yield by GC versus internal standard (Scheme 3).

2.2. X-ray crystal structure of **2**·CH_2Cl_2

In the solid state, complex **2** adopts a distorted linear conformation with a P–Au–alkene$_{(cent)}$ angle of 160.2° and with the diene ligand bound unsymmetrically to gold through a shorter Au–C1 and a longer Au–C2 interaction ($\Delta d = 0.188$ Å) (Fig. 1). The coordinated C1=C2 bond of the diene is elongated relative to the uncomplexed C3=C4 bond ($\Delta d = 0.039$ Å) and the diene adopts a



Scheme 1.

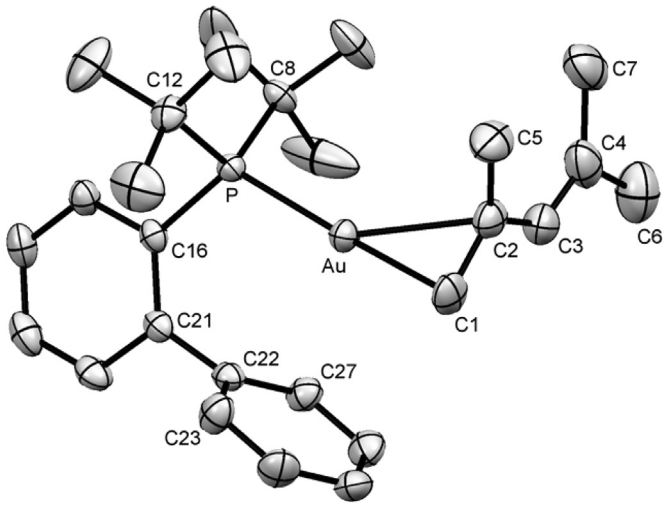
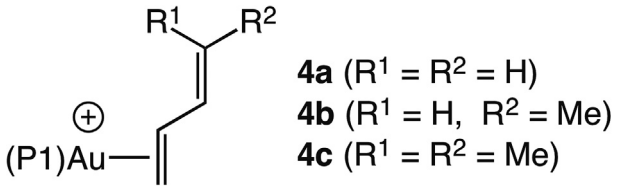


Fig. 1. ORTEP drawing of 2•CH_2Cl_2. Ellipsoids are shown at the 50% probability level with counterion, solvent, and hydrogen atoms omitted for clarity. Selected bond distances (Å), bond angles (deg), and torsion angles (deg): Au–C1 = 2.202(4), Au–C2 = 2.390(4), C1–C2 = 1.395(7), C2–C3 = 1.458(7), C3–C4 = 1.356(7), C2–C5 = 1.505(6), Au–arene$_{(plane)}$ = 3.04, Au–C22 = 3.08, P–Au–C1=C2(cent) = 160.2, C1–C2–C3 = 117.6(4), C1–C2–C3–C4 = 175.8(5), C16–C21–C22–C23 = 101.2(5), C16–C21–C22–C27 = 85.0(5).

near planar *s-trans* configuration with a C1–C2–C3–C4 dihedral angle of 175.8°. The diene ligand is positioned such that the plane defined by Au–C1–C2 is rotated ~35° relative to the plane defined by Au–P–C16 with the C=CMe_2 group directed away from the protruding phenyl ring. The protruding phenyl group of the *o*-biphenylphosphine moiety is nearly perpendicular to the P-bound aryl ring with a C16–C21–C22–C27 dihedral angle of 85°. The distance between the gold atom and the ipso carbon of the protruding phenyl ring (C22) is 3.08 Å, suggesting the presence of a weak Ar–arene interaction, as has been previously noted for cationic gold complexes containing the **P1** ligand [10–12,19].

Comparison of the structure of **2** to the structures of the related gold π-diene complexes **4** that contain a **P1** ligand and a C1, C2-unsubstituted diene ligand provides insight into the effect of the C2 methyl substituent of **2** on the gold π-diene interaction (Table 1) [12]. The most notable difference between **2** and complexes **4** is the more pronounced slippage of gold toward the terminal C1 atom of the diene ligand. This slippage is evidenced both in the greater deviation from linearity of the P–Au–C=$C_{(cent)}$ angle of **2** relative to **4** and in the greater difference between the Au–C1 and Au–C2 distances (Δ*d*) of **2** relative to those of **4**. Also worth noting is the orientation of the coordinated C1=C2 bond in complexes **4**, as defined by the Au–C1–C2 plane, relative to the **P1** ligand, as defined by Au–P–$C_{(ipso)}$ plane, ranges from 12° in the case of **4a** to ~70–75° in the case of **4c** with **2** (35°) falling between these extremes. As we have previously noted in the context of gold π-alkene complexes [15,20], the rotational orientation of the C=C bond relative to the (L)Au fragment appears to be controlled primarily by steric interactions without any notable electronic preference.

Table 1
Comparison of geometric parameters defining the slippage and rotational orientation of the diene ligand of complexes **2**·CH_2Cl_2, **4a**, **4b**, and **4c**.



Compound	Δ*d* Au–C1/Au–C2 (Å)	P–Au–(C1=C2)$_{cent}$ (deg)	(Au–P–C_{ipso})–(Au–C1–C2) (deg)
2	0.19	160	35
4a[a,b]	0.09	167	20
4b[a,b]	0.11	169	13
4c[a]	0.08	165.5	72

[a] Taken from reference [12].
[b] Average of two disordered molecules.

3. Conclusion

We have reported the isomerization of the cationic gold tetramethylallene complex [(**P1**)Au(η^2-Me_2C=C=CMe_2)]$^+$ SbF_6^- (**1**) via formal 1,3-hydrogen migration to form the conjugated diene complex {(**P1**)Au[η^2-H_2C=C(Me)–C(H)=CMe_2]}$^+$ SbF_6^- (**2**), which occurs at or below room temperature in the absence of any apparent Brønsted base. These observations establish the ability of cationic gold(I) complexes to mediate the conversion of aliphatic allenes to conjugated dienes and further reveals significant π-acidity of the cationic, twelve electron (**P1**)Au$^+$ fragment [15]. We also report the X-ray crystal structure of gold π-diene complex **2**, which was distinguished from related gold diene complexes **4** by the more pronounced slippage of the coordinated C1=C2 bond due to the C2 methyl group.

4. Experimental

4.1. {[P(t-Bu)$_2$o-biphenyl]Au[η^2-H_2C=C(Me)C(H)=CMe_2]}$^+$ SbF_6^-(**2**)

Slow vapor diffusion of hexanes (15 mL) into a solution of **1** (62 mg, 7.5 × 10^{-2} mmol, 75 mM) in CH_2Cl_2 (1 mL) at 4 °C for 36 h formed colorless prismatic crystals of **2**·CH_2Cl_2 which were separated from the mother liquor, rinsed with cold hexanes (3 × 5 mL), and dried to give **2**·CH_2Cl_2 (57 mg, 92%). ^{1}H NMR: δ 7.93–7.88 (m, 1 H), 7.65–7.55 (m, 5 H), 7.28–7.20 (m, 3 H), 5.84 (s, 1 H), 3.89 (d, *J* = 3.5 Hz, 1 H), 3.81 (d, *J* = 4.0 Hz, 1 H), 2.27 (s, 3 H), 1.96 (s, 3 H), 1.92 (s, 3 H), 1.37 (d, *J* = 16.5 Hz, 18 H). ^{31}P NMR: δ 67.5. This spectral data is consistent with the published spectroscopy of **2** [12].

4.2. In situ conversion of **1** to **2**

A solution of **1** (25 mg, 3.0 × 10^{-2} mmol, 5.5 × 10^{-2} mM) and 1,3-dimethoxybenzene (1.0 μL, 7.6 μmol; internal standard) in CD_2Cl_2 (0.55 mL) was monitored periodically by ^{1}H and ^{31}P NMR spectroscopy at 25 °C. The relative concentrations of **1** and **2** were determined by integrating the methyl resonance of **1** at δ 1.93 and the olefinic resonances of **2** at δ 3.89 (d, *J* = 3.5 Hz, 1H) and 3.81 (d, *J* = 4.0 Hz, 1H) relative to the methoxy resonance of 1,3-dimethoxybenzene at δ 3.71 in the ^{1}H NMR spectrum and by integrating the phosphorous resonances of **1** (δ 66.7) and **2** (δ 67.5) in the ^{31}P NMR spectrum. *in situ* Analysis of the conversion of **1** (55 mM in CD_2Cl_2) to **2** in the presence of 2,4-dimethyl-2,3-pentadiene (55 mM) and the reaction of **1** (55 mM in CD_2Cl_2) with triethylamine (55 mM) were performed employing similar procedures.

4.3. X-ray data crystal structure of **2**·CH_2Cl_2

A crystal of **2**·CH_2Cl_2 was mounted on a Mitegen polyimide micromount with a small amount of Paratone N oil and analyzed on a Bruker-Nonius Kappa Axis X8 Apex2 diffractometer at 110 K. The unit cell dimensions were determined from a symmetry constrained fit of 9966 reflections with 4.36° < 2θ < 57.98°. The data collection strategy was a number of ω and φ scans, collected up to

66.36° (2θ). The frame integration was performed using SAINT [21]. The resulting data was scaled and corrected for absorption using a multi-scan averaging of symmetry equivalent data using SADABS [22]. The structure was solved by direct methods using the XS program [23]. All non-hydrogen atoms were obtained from the initial solution. The hydrogen atoms were introduced at idealized positions and were allowed to ride on the parent atom. The structural model was fit to the data using full matrix least-squares based on F2. The calculated structure factors included corrections for anomalous dispersion from the usual tabulation. The structure was refined using the XL program from SHELXTL graphic plots were produced using the NRCVAX crystallographic program suite [24].

Crystal Data for **2**·CH_2Cl_2: CCDC #978518, $C_{28}H_{41}AuCl_2F_6PSb$, M = 912.19, orthorhombic, space group Pbca, a = 18.6822(7), b = 18.6579(7), c = 18.8212(7) Å, V = 6560.5(4) Å^3, Z = 8, D_c = 1.847 (g/cm^3), μ = 5.554−cm^{-1}, reflections measured 254666, 12540 unique (R_{merge} = 0.0645) which were used in all calculations. The final R_1 was 0.0647 and the final wR_2 was 0.0951 (all data).

Acknowledgments

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Appendix A. Supplementary material

CCDC 978518 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

References

[1] (a) S. Tsuboi, T. Masuda, A. Takeda, J. Org. Chem. 47 (1982) 4478–4482;
(b) B.M. Trost, U. Kazmaier, J. Am. Chem. Soc. 114 (1992) 7933–7935;
(c) E. Hayashi, R.P. Hsung, J.B. Feltenberger, A.G. Lohse, Org. Lett. 11 (2009) 2125–2128;
(d) R. Sanz, D. Miguel, A. Martinez, M. Gohain, P. Garcia-Garcia, M.A. Fernández-Rodriguez, E. Álvarez, F. Rodriguez, Eur. J. Org. Chem. (2010) 7027–7039;
(e) R. Hayashi, J.B. Feltenberger, A.G. Lohse, M.C. Walton, R.P. Hsung, Beilstein J. Org. Chem. 7 (2011) 410–420;
(f) A.K. Buzas, F.M. Istrate, F. Gagosz, Org. Lett. 9 (2007) 985–988;
(g) A.S. Dudnik, T. Schwier, V. Gevorgyan, Tetrahedron 65 (2009) 1859–1870;
(h) X. Zhang, C. Fu, S. Ma, Org. Lett. 13 (2011) 1920–1923;
(i) M.E. Krafft, K.M. Hallal, D.V. Vidahani, J.W. Cran, Org. Biomol. Chem. 9 (2011) 7535–7538;
(j) W. Peng, S. Zhu, Tetrahedron 59 (2003) 4641–4649.
[2] For the thermal isomerization of alkylidecyclopropanes: (a) J.K. Crandall, D.R. Paulson, J. Am. Chem. Soc. 88 (1966) 4302–4303;
(b) D.R. Paulson, J.K. Crandall, C.A. Bunnell, J. Org. Chem. 35 (1970) 3708–3714;
(c) M.E. Hendrick, J.A. Hardie, M. Jones, J. Org. Chem. 36 (1971) 3061–3062;
(d) R. Bloch, P.L. Perchec, J.-M. Conia, Angew. Chem. Int. Ed. Engl. 9 (1970) 798–799;
(e) T.B. Patrick, E.C. Haynie, W.J. Probst, Tetrahedron Lett. 27 (1971) 423–424;
(f) W. Lenk, H. Hopf, Tetrahedron Lett. 23 (1982) 4073–4076.
[3] (a) F. Jensen, J. Am. Chem. Soc. 117 (1995) 7487–7492;
(b) N. Krause, A.S.K. Hashmi (Eds.), Modern Allene Chemistry, Wiley-VCH, Weinheim, Germany, 2004.
[4] H. Meier, M. Schmitt, Tetrahedron Lett. 30 (1989) 5873–5876.
[5] M. Al-Masum, Y. Yamamoto, J. Am. Chem. Soc. 120 (1998) 3809–3810.
[6] C.-M. Ting, Y.-L. Hsu, R.-S. Liu, Chem. Commun. 48 (2012) 6577–6579.
[7] R. Ben-Shoshan, R. Pettit, J. Am. Chem. Soc. 89 (1967) 2231–2232.
[8] (a) D.J. Gorin, B.D. Sherry, F.D. Toste, Chem. Rev. 108 (2008) 3351–3378;
(b) A. Arcadi, Chem. Rev. 108 (2008) 3266–3325;
(c) Z. Li, C. Brouwer, C. He, Chem. Rev. 108 (2008) 3239–3265;
(d) P.Y. Toullec, V. Michelet, Top. Curr. Chem. 302 (2011) 31–80;
(e) A. Fürstner, P.W. Davies, Angew. Chem. Int. Ed. 46 (2007) 3410–3449;
(f) E. Jiménez-Núñez, A.M. Echavarren, Chem. Rev. 108 (2008) 3326–3350;
(g) A. Corma, A. Leyva-Pérez, M.J. Sabater, Chem. Rev. 111 (2011) 1657–1712;
(h) N. Krause, C. Winter, Chem. Rev. 111 (2011) 1994–2009;
(i) M. Rudolph, A.S.K. Hashmi, Chem. Soc. Rev. 41 (2012) 2448–2462.
[9] (a) R.E.M. Brooner, R.A. Widenhoefer, Angew. Chem. Int. Ed. 52 (2013) 11714–11724;
(b) H. Schmidbaur, A. Schier, Organometallics 29 (2010) 2–23.
[10] T.J. Brown, A. Sugie, M.G. Dickens, R.A. Widenhoefer, Chem. Eur. J. 18 (2012) 6959–6971.
[11] T.J. Brown, A. Sugie, M.G. Dickens, R.A. Widenhoefer, Organometallics 29 (2010) 4207–4209.
[12] R.E.M. Brooner, R.A. Widenhoefer, Organometallics 30 (2011) 3182–3193.
[13] (a) R.A. Sanguramath, T.N. Hooper, C.P. Butts, M. Green, J.E. McGrady, C.A. Russell, Angew. Chem. Int. Ed. 50 (2011) 7592–7595;
(b) R.A. Sanguramath, S.K. Patra, M. Green, C.A. Russell, Chem. Commun. 48 (2012) 1060–1062.
[14] The enthalpy of reaction of the conversion of 2,4-dimethyl-2,3-pentadiene to 2,4-dimethyl-1,3-pentadiene was estimated to be −7.8 kcal/mol based on the calculated (PM3/Spartan) heats of formation. The binding affinity of 2,4-dimethyl-1,3-pentadiene to the (P1)Au$^+$ fragment was calculated to exceed that of 2,4-dimethyl-2,3-pentadiene by a factor of ~7 (−60 °C, CD_2Cl_2) from the known equilibrium binding affinities of 2,4-dimethyl-1,3-pentadiene relative to isobutylene (K_{eq} = 0.9), [12] isobutylene relative to $NCAr_F$ [$NCAr_F$ = 3,5-$NCC_6H_3(CF_3)_2$] (K_{eq} = 130), [15] and 2,4-dimethyl-2,3-pentadiene relative to $NCAr_F$ (K_{eq} = 17) [11].
[15] T.J. Brown, M.G. Dickens, R.A. Widenhoefer, Chem. Commun. (2009) 6451–6453.
[16] A. Zhdanko, M. Ströbele, M.E. Maier, Chem. Eur. J. 18 (2012) 14732–14744.
[17] A. Basak, K. Chakrabarty, A. Ghosh, G.K. Das, J. Org. Chem. 78 (2013) 9715–9724.
[18] T.J. Brown, R.A. Widenhoefer, Organometallics 30 (2011) 6003–6009.
[19] (a) E. Herrero-Gómez, C. Nieto-Oberhuber, S. López, J. Benet-Buchholz, A.M. Echavarren, Angew. Chem. Int. Ed. 45 (2006) 5455–5459;
(b) P. Pérez-Galán, N. Delpont, E. Herrero-Gómez, F. Maseras, A.M. Echavarren, Chem. Eur. J. 16 (2010) 5324–5332;
(c) D.V. Partyka, T.J. Robilotto, M. Zeller, A.D. Hunter, T.G. Gray, Organometallics 27 (2008) 28–32;
(d) D.V. Partyka, J.B. Updegraff, M. Zeller, A.D. Hunter, T.G. Gray, Organometallics 28 (2009) 1666–1674;
(e) F.-B. Xu, Q.-S. Li, L.-Z. Wu, X.-B. Leng, Z.-C. Li, X.-S. Zeng, Y.L. Chow, Z.-Z. Zhang, Organometallics 22 (2003) 633–640;
(f) Q.-S. Li, C.-Q. Wang, R.-Y. Zou, F.-B. Xu, H.-B. Song, X.-J. Wan, Z.-Z. Zhang, Inorg. Chem. 45 (2006) 1888–1890.
[20] T.J. Brown, M.G. Dickens, R.A. Widenhoefer, J. Am. Chem. Soc. 131 (2009) 6350–6351.
[21] Bruker-Nonius, SAINT Version 2009.9, Bruker-Nonius, Madison, WI 53711, USA, 2009.
[22] Bruker-Nonius, SADABS Version 2009.9, Bruker-Nonius, Madison, WI 53711, USA, 2009.
[23] Bruker-AXS, XS Version 2009.9, Bruker-AXS, Madison, WI 53711, USA, 2009.
[24] Bruker-AXS, XL Version 2009.9, Bruker-AXS, Madison, WI 53711, USA, 2009.