Use of ionic liquids in the platinum- and gold-catalyzed cycloisomerization of enyne systems

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ABSTRACT

The platinum- and gold-catalyzed cycloisomerization of enyne systems has been carried out in various ionic liquids (ILs). In some cases, better selectivities and shorter reaction times have been observed compared to conventional conditions.

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1. Introduction

Cycloisomerization of polyunsaturated acyclic molecules is highly desirable due to the increase of complexity it produces in the final product from generally readily accessible precursors [1,2]. Among the various precursors, the enyne moiety associated with electrophilic metals species such as $PtCl_2$, $AuCl_3$, and AuCl derivatives allowed the preparation of a large variety of polycyclic compounds [3–11].

For instance, we [12–17] and others [18–30] have shown that different acyl groups at a propargylic position of an enyne can migrate in a 1,2-fashion with concomitant intramolecular cyclopropanation onto the ene part of the substrate. We demonstrated that acyclic propargylic enyne acetates such as **1** are useful precursors of bicyclic compounds **2** [12,15,31]. Taking into account that charged intermediates are generally involved in these processes, we have been interested in the use of alternative solvents such as ionic liquids (ILs) [32]. Indeed, such organic salts have been largely used over the last two decades due to their original physicochemical properties such as high thermal stability, low vapor pressure, non-inflammability and polar properties [33–35].

Generally speaking, ILs are one of the leading alternatives to the traditional organic solvents [36,37].

Catalytic reactions performed in ILs have been the subject of intense research. An attractive feature is that most of the active complexes are stable in such media allowing the possibility to reuse the catalyst several times with negligible decrease in activity [38–44]. In comparison to palladium-, rhodium- and ruthenium-catalyzed transformations, which have been extensively studied, platinum and gold complexes have been much less used in association with molten salts [45] or ILs. Doherty et al. reported efficient enantioselective platinum-catalyzed carbonyl ene alkylations [46]. Hydrosilylations of alkenes [47,48] or direct oxidation of methane [49] have been performed into ionic liquids. The first thiazolium gold(III) compound that qualifies as an ionic liquid has been prepared in 2002. In association with other imidazolium ILs, this complex as well as other gold salts were efficient catalysts for the hydration of phenyl acetylene [50]. Electrophilic activation of the alkyne function of an enyne precursor to give skeletal rearrangement products could also be accomplished through $PtCl_2$ catalysis in ILs, as disclosed by Chatani in 2004 [51]. In the same vein, gold-catalyzed intramolecular nucleophilic attacks of oxygen or nitrogen atoms onto alkynyl functions have been reported to proceed in good to excellent yields in various ILs giving the corresponding furans and indoles, respectively [52,53]. Finally, a recent report by Krause on the Au-catalyzed synthesis of 2,5-dihydrofurans from allene precursors in ILs prompted us to report our own findings in this area of investigation [54].

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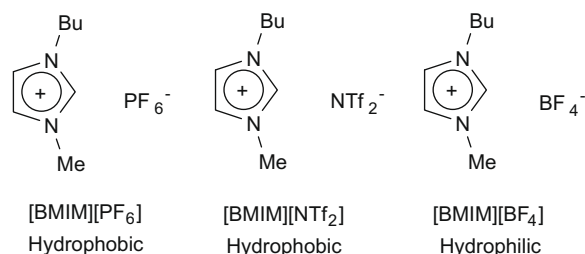
Fig. 1. Structure of ionic liquids.

2. Results and discussion

Thus, we started our study with precursor **1** and used the following ILs (Fig. 1). We initially investigated the platinum(II) catalysis. Among all the solvents used (Table 1, entries 2–4), [BMIM][NTf$_2$] proved to be the most reliable since 99% yield of a 9:1 mixture of **2a** and **2b** was obtained while the other ones gave more complex intractable mixtures.

Gold catalysis provided contrasted results. A more rapid and selective reaction than the regular CH$_2$Cl$_2$ solution one (entry 5) was observed with the use of AuCl$_3$ in [BMIM][PF$_6$] and in [BMIM][NTf$_2$] (entries 6 and 7) giving a mixture of only **2a** and **2b**. However, in sharp contrast, but not unexpectedly, methylketone **2d** was the major product when the hydrophilic [55–57] [BMIM][BF$_4$] IL was used (entry 8). The same trend was observed with the use of AuCl. A fast and more selective reaction was observed in [BMIM][PF$_6$] or [BMIM][NTf$_2$] (entries 10 and 11) and the [BMIM][BF$_4$] IL resulted also in the major formation of the hydration product. Finally, cationic gold(I) catalysis proved to be in our hands inapplicable in IL (see entries 14 and 15) yielding in each case a lot of hydration product.

To draw some simple conclusions from this study, it appears that hydrophobic IL can provide good yields of cycloisomerization products, generally in an altered selectivity compared to the CH$_2$Cl$_2$ solution reaction. An obvious benefit from the utilization of IL notably with gold catalysis resides in the rapidity of the reaction: 1 min instead of over 1 h (entry 5 vs. 6)! This probably can be rationalized by a concentration effect since these reactions are conducted at 0.57 M in IL while they are generally run at much lesser concentration in typical solution conditions (0.025 M). Consistent with this, it has been reported that platinum-catalyzed cycloizomerisation reactions in neat conditions allowed to significantly decrease the reaction time [17]. Importantly this finding suggests high-throughput procedures can certainly be devised.

In the same vein, we tested the recyclability of the catalyst solution. As shown in Table 2, up to 5 cycles could be operated that still gave a decent yield (80%) of the **2a:2b** mixture at the fifth iteration (Table 2).

We also examined other substrates in order to test the generality of the acetate migration based cycloisomerization reaction. Thus, precursors **3** and **5** provided the expected polycyclic products **4** and **6** in fair yields, but not better than the typical solution reactions (Scheme 1).

Thus, it was clear from these results that the formation of cyclopropane, presumably involving a metalla-carbene intermediate, was possible. We then wanted to look at the possibility of having a bis-cyclopropanation reactivity from a dienyne precursor as

Table 2
Recycling experiment of platinum-catalyzed cycloisomerization in ionic liquids.

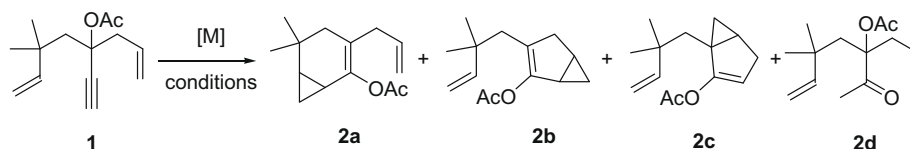| Cycle[a]	2a:2b[b]	Yield (%)
1	90:10	99
2	90:10	96
3	90:10	94
4	88:12	90[c]
5	87:13	80[d]

[a] Run with PtCl$_2$ in [BMIM][NTf$_2$], 1 h at 80 °C.
[b] Determined by ^{1}H NMR.
[c] 7% S.M. recovered.
[d] 15% S.M. recovered.

Table 1
Distribution of O-acyl migration products in usual solvents and ionic liquids.

Entry	[M]	Conditions[a]	2a:2b:2c:2d[b]	Yield (%)
1	PtCl$_2$	Toluene, 80 °C, 2 h	90:10:0:0	90[c]
2	PtCl$_2$	[BMIM][PF$_6$], 80 °C, 1 h	Mixture[d]	–
3	PtCl$_2$	[BMIM][NTf$_2$], 80 °C, 1 h	90:10:0:0	99
4	PtCl$_2$	[BMIM][BF$_4$], 80 °C, 1 h	Mixture[d]	–
5	AuCl$_3$	CH$_2$Cl$_2$, r.t., 1.5 h	50:38:12:0	88
6	AuCl$_3$	[BMIM][PF$_6$], r.t., 1 min	80:20:0:0	92
7	AuCl$_3$	[BMIM][NTf$_2$], r.t., 1 min	80:20:0:0	91
8	AuCl$_3$	[BMIM][BF$_4$], r.t., 1.5 h	40:10:0:50	80
9	AuCl	CH$_2$Cl$_2$, r.t., 20 min	54:35:11:0	80
10	AuCl	[BMIM][PF$_6$], r.t., 1 min	80:20:0:0	95
11	AuCl	[BMIM][NTf$_2$], r.t., 1 min	80:20:0:0	97
12	AuCl	[BMIM][BF$_4$], r.t., 1.5 h	40:10:0:50	92
13	AuClPPh$_3$/AgPF$_6$	CH$_2$Cl$_2$, r.t., 20 min	77:0:23:0	75
14	AuClPPh$_3$/AgPF$_6$	[BMIM][PF$_6$], r.t., 20 min	20:0:0:80	65
15	AuClPPh$_3$/AgPF$_6$	[BMIM][NTf$_2$], r.t., 20 min	20:0:0:80	86

[a] General procedure: dienyne **1** (0.4 mmol), PtCl$_2$ (5 mol%) or [Au] (2 mol%), ionic liquid (0.7 mL).
[b] Determined by ^{1}H NMR.
[c] See Ref. [15].
[d] A complex and intractable mixture of compounds including products **2** was obtained.
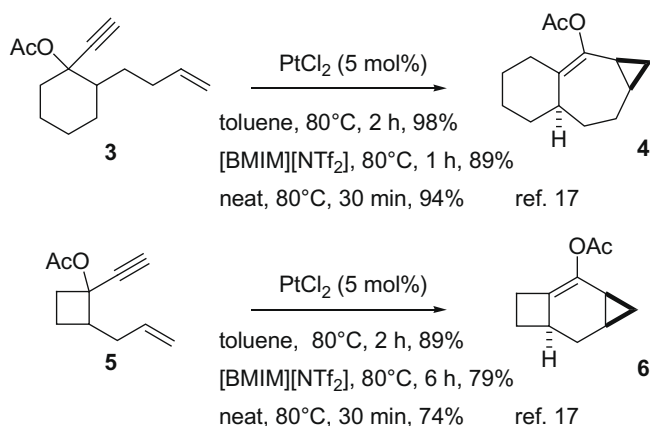
Scheme 1. Formation of polycyclic derivatives by platinum-catalyzed acetate migration.

experienced with **7** to give **8** with PtCl₂ or PtCl₄ in solution (Table 3) [12,15]. We also checked the reactivity of this substrate with gold catalysts and we observed shorter reaction times even at lower temperature. Interestingly, while AuBr₃ and AuCl gave similar results with the main formation of **8**, cationic gold catalyst AuClPPh₃/AgSbF₆ marked a significant contrast in the evolution of the system since cyclohexadienic compound **9** was the main product (58% yield). This type of product has been interpreted by Echavarren as an *endo*-cleavage type product [58,59,15]. Aromatic products (**10a** and **10b**) originating from a loss of MeOH were also isolated as an inseparable mixture (Table 3, entry 5). We suspected that the formation of these products could be influenced by the amount of precatalyst mixture.

Indeed, with 20 mol% of AuClPPh₃/AgSbF₆, a mixture of **10a** and **10b** was obtained in 19% yield accompanied with 42% of a new product, the cyclohexenone **11** (Scheme 2). The latter presumably originates from a proton or metal-catalyzed [3,3]-sigmatropic rearrangement of **9** followed by an hydrolysis of the enol-ether intermediate. A loss of methanol would lead to the formation of the conjugated triene **12**. cationic [1,2] methyl shifts and further aromatization deliver the corresponding allyl-pseudocumene derivatives **10a** and **10b** [60,61].

We then studied the metal-catalyzed transformations of **7** in ILs. We focus on PtCl₂, AuBr₃ and AuCl since cationic gold(I) proved to be touchy in our previous study and here also the solution

chemistry was not particularly encouraging since several products were formed (see above). We obtained the following results (Table 4).

With platinum chloride in ionic liquids, all the attempts exhibited very slow reactions and a majority of starting material was recovered after many hours. We tried to force the reactions conditions but the decompositions of the starting material was observed. Interestingly, with AuCl and AuBr₃, we obtained mixtures which recall the outcomes of reactions with cationic gold(I) catalysis, and it is worthy of note that the formation of tetracyclic derivative **8** is poorly operative.

In conclusion, we have examined well-established platinum(II), gold(III) and gold(I)-catalyzed cycloisomerization reactions in ILs. We could obtain similar results to the typical solution reactions in the case of the migration of acetate based cycloisomerizations. In some cases, much shorter reaction times were observed. Interestingly, cationic gold(I) catalysis does not appear to be viable in this type of reaction medium. With the dienyne precursor **7**, it is interesting to note that the formation of the expected tetracyclic product **8** was not efficient in ILs. Instead, the IL reactions provided somehow the same products we obtained with the gold(I) cationic catalysis which suggests an important role of the reaction medium for this transformation.

All these findings are of interest and they confirm that ILs can be advantageous since high-throughput procedures can be at hand after optimization, and as well as opportunities of new reactivity with already known precursors. Such particular reactions conditions could be of a great help for mechanistic investigations in using the property of ionic liquid to promote charges formations.

3. Experimental

3.1. General remarks

All reactions were run under an argon or a nitrogen atmosphere in anhydrous solvents and flame-dried flask. Toluene and dichloromethane were distilled from CaH₂. Thin-layer chromatography (TLC) was performed on Merck 60 F254 silica gel. Merck Gerudan SI 60 Å silica gel (35–70 μ) was used for column chromatography. NMR spectra were recorded at room temperature on a 400 MHz Bruker ARX400 spectrometer. Chemical shifts are given in parts per million, referenced to the residual proton resonance of the solvents (=7.26 ppm for CDCl₃) or to the residual carbon resonance of the solvent (=77.16 ppm for CDCl₃). High-resolution mass spectra (HRMS) were measured by the Service de Spectrométrie de Masse de l'Université Pierre et Marie Curie-Paris 6. Infrared (IR) spectra were recorded on a Bruker Tensor 27 spectrometer. Gold and platinum catalysts were purchased from Strem. Cycloisomerizations were performed in freshly degassed (2–3 freeze–pump–thaw cycles or 30 min argon bubbling) solvents. [BMIM][PF₆], [BMIM][NTf₂] and [BMIM][BF₄] were purchased from Solvionic and kept 4 h under vacuum (10⁻² bar) before use. The preparation of the dienyne substrates **1** and **5** have been reported [15], and the corresponding cycloisomerization products **2** have been characterized [31]. The enynes **3** and **5** have been prepared and the cycloisomerization products have been characterized [17].

3.2. General procedure for the cycloisomerization in usual solvent

PtCl₂ (5 mol%), AuCl (2 mol%), AuCl₃ (2 mol%) or AuClPPh₃ (2 mol%) and AgSbF₆ (2 mol%) were introduced in a round bottom flask under an argon atmosphere. Anhydrous toluene or CH₂Cl₂ (0.025 M) was then added. Dienyne (0.4 mmol) was introduced and the reaction mixture was stirred at the appropriate temperature (for 1–2 h at 80 °C with PtCl₂, 1–90 min at rt with gold

Table 3
Cycloisomerization of dienyne **7** in usual solvents.

Entry	[M]	Conditions	**8**[c] (%)	**9**[c] (%)
1	PtCl₂[a]	Toluene, 80 °C, 2 h	73[e]	–
2	PtCl₄[a]	Toluene, 80 °C, 2 h	77[e]	–
3	AuBr₃[b]	CH₂Cl₂, r.t., 20 min	69	–
4	AuCl[b]	CH₂Cl₂, r.t., 20 min	72	–
5	AuClPPh₃/AgSbF₆[a]	CH₂Cl₂, r.t., 20 min	22	58[d]

[a] 5 mol% of catalyst.
[b] 2 mol% of catalyst.
[c] Isolated yields.
[d] 5% of **10a** and **10b** have been isolated.
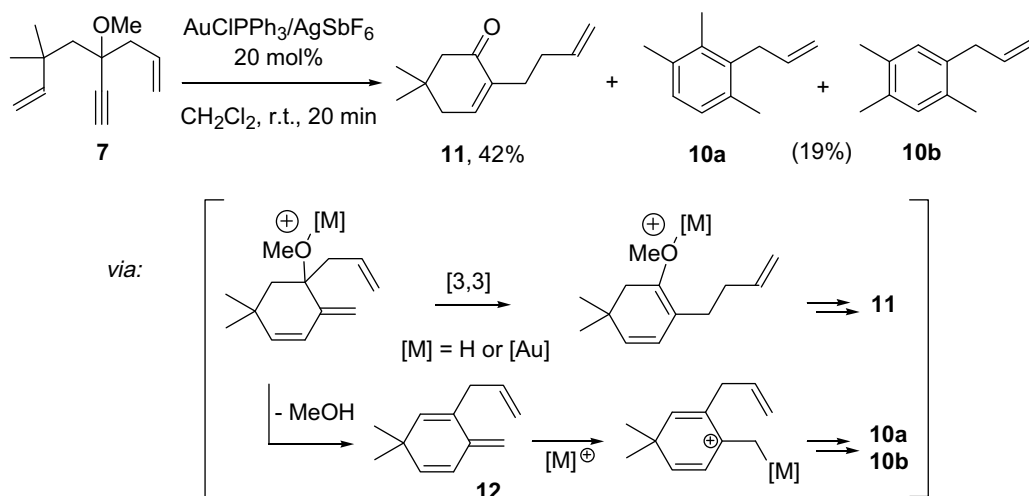[e] See Ref. [15].

Scheme 2. Behavior of dienyne **7** with gold(I) catalyst.

Table 4
Behavior of dienyne **7** with gold(I) catalyst in ionic liquids.

Entry	[Au]	Conditions	**7:8:9:10**	Yield (%)
1	AuBr$_3$	[BMIM][PF$_6$], 2.5 h	32:10:58:0	69
2	AuBr$_3$	[BMIM][NTf$_2$], 2.5 h	48:0:52:0	69
3	AuCl	[BMIM][PF$_6$], 2.5 h	50:19:31:0	73
4	AuCl	[BMIM][NTf$_2$], 2.5 h	31:10:24:35	81

catalysts) and was monitored by TLC. After completion, the solution was filtered through a pad of silica and the pad was washed with diethylether. The solvent was removed under reduced pressure and the crude mixture was purified by flash chromatography.

3.3. General procedure for the cycloisomerization in ionic liquid

PtCl$_2$ (5 mol%) or gold salts (2 mol%) was added to ionic liquid (0.7 mL). The mixture was stirred for 5 min and warmed to the desired temperature (80 °C for PtCl$_2$, r.t. for gold salts) and dienyne (0.4 mmol) was added. The mixture was stirred at this temperature for the indicated time and extracted with Et$_2$O (3 × 1 mL). The solvent was evaporated under reduced pressure and the crude mixture was purified by flash chromatography.

3.4. Catalyst recycling

Same procedure with dry cyclohexane (5 × 1 mL) as extracting solvent. The ionic liquid media was taken up in benzene, stirred 5 min and the solvent was evaporated. The dienyne **1** was then added to this mixture for the next run.

Ketone **2d**: Colorless oil. ^{1}H NMR (CDCl$_3$, 400 MHz) δ 5.72 (dd, J = 17.6, 10.8 Hz, 1H), 5.53 (ddt, J = 17.6, 10.4, 7.6 Hz, 1H), 5.04 (m, 2H), 4.84 (m, 2H), 2.96 (dd, J = 14.4, 7.6 Hz, 1H), 2.62 (dd, J = 14.4, 7.6 Hz, 1H), 2.29 (d, J = 15.2 Hz, 1H), 2.15 (s, 3H), 2.09 (d, J = 15.2 Hz, 1H), 2.04 (s, 3H), 1.02 (s, 3H), 0.96 (s, 3H). ^{13}C NMR (CDCl$_3$, 100 MHz) δ 209.3 (C), 170.0 (C), 147.9 (CH), 131.2 (CH), 119.7 (CH$_2$), 110.5 (CH$_2$), 90.5 (C), 45.7 (CH$_2$), 41.2 (CH$_2$), 36.6 (C), 28.3 (CH$_3$), 28.1 (CH$_3$), 28.0 (CH$_3$), 22.2 (CH$_3$). IR (neat) 3081,

2962, 2933, 2874, 1740, 1720, 1236 cm^{-1}. HRMS Calc. for C$_{14}$H$_{22}$O$_3$Na [M+Na]: 261.14612; Found: 261.14603.

Cyclohexene **9**: Colorless oil. ^{1}H NMR (CDCl$_3$, 400 MHz) δ 5.94 (d, J = 9.8 Hz, 1H), 5.84 (m, 1H), 5.51 (d, J = 9.8 Hz, 1H), 5.11–5.05 (overlap, 4H), 3.09 (s, 3H), 2.58 (dd, J = 14.6, 6.4 Hz, 1H), 2.36 (dd, J = 14.6, 6.8 Hz, 1H), 1.85 (d, J = 14.0, 1H), 1.51 (d, J = 14.0 Hz, 1H), 1.14 (s, 3H), 1.02 (s, 3H). ^{13}C NMR (CDCl$_3$, 100 MHz) δ 142.3 (C), 139.6 (CH), 134.6 (CH), 125.5 (CH), 117.6 (CH$_2$), 113.0 (CH$_2$), 76.5 (C), 49.5 (CH$_3$), 44.3 (CH$_2$), 40.4 (CH$_2$), 33.4 (C), 31.5 (CH$_3$), 30.6 (CH$_3$). IR (neat) 2933, 1460, 1079 cm^{-1}. HRMS Calc. for C$_{13}$H$_{20}$ONa [M+Na]: 215.14064; Found: 215.14032.

Cyclohexenone **11**: Colorless oil. ^{1}H NMR (CDCl$_3$, 400 MHz) δ 6.56 (t, J = 4.4 Hz, 1H), 5.77 (ddt, J = 20.2, 16.8, 6.4 Hz, 1H), 4.97 (m, 2H), 2.31–2.15 (overlap, 8H), 1.02 (s, 6H). ^{13}C NMR (CDCl$_3$, 100 MHz) δ 199.7 (C), 143.4 (CH), 138.4 (CH), 138.2 (C), 115.1 (CH$_2$), 52.3 (CH$_2$), 40.3 (CH$_2$), 34.2 (C), 32.9 (CH$_2$), 28.9 (CH$_2$), 28.4 (2 CH$_3$). IR (neat) 2923, 1675, 1464 cm^{-1}. HRMS Calc. for C$_{12}$H$_{19}$O [M+H]: 179.14304; Found: 179.14297.

Allyl-trimethylbenzene derivatives **10a** and **10b**: Inseparable mixture 1.0/1.7 (m)/(M). Colorless oil. ^{1}H NMR (CDCl$_3$, 400 MHz) δ 6.96–6.93 (overlap, 4H, M and m), 6.01–5.88 (overlap, 2H, M and m), 5.05–5.02 (overlap, 3H, M and m), 4.88 (ddd, J = 17.1, 3.8, 1.8 Hz, 1H, m), 3.44 (dt, J = 5.7, 1.8 Hz, 2H, m), 3.33 (br. d, J = 6.7 Hz, 2H, M), 2.29 (s, 3H, m), 2.28 (s, 3H, m), 2.25–2.21(overlap, 12H, M and m). ^{13}C NMR (CDCl$_3$, 100 MHz) δ 137.2 (CH, M), 136.1 (Carom, m), 135.8 (CH, m), 135.5 (Carom, M), 135.3 (Carom, m), 134.5 (Carom, m), 134.4 (Carom, M), 134.3 (Carom, m), 134.1 (Carom, M), 133.6 (Carom, M), 131.7 (CH, M), 130.7 (CH, m), 127.9 (CH, M), 127.5 (CH, m), 115.4 (CH$_2$, M), 115.0 (CH$_2$, m), 37.5 (CH$_2$, M), 34.1 (CH$_2$, m), 20.9 (CH$_3$, m), 20.1(CH$_3$, m), 19.3 (2 CH$_3$, M), 18.9 (CH$_3$, M), 15.6 (CH$_3$, m). IR (neat) 2923, 2853, 1461 cm^{-1}. HRMS Calc. for C$_{12}$H$_{17}$ [M+H]: 161.13248; Found: 161.13224.

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