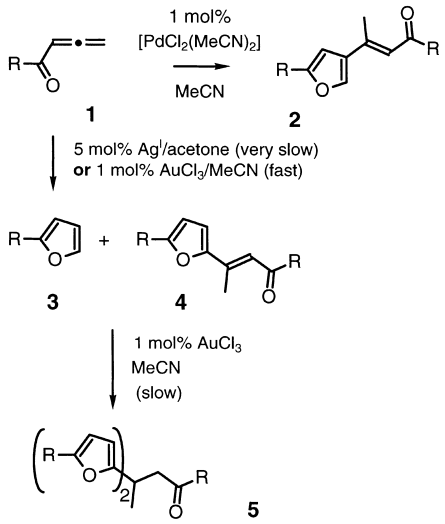
[9] S. Pitsch, C. Spinner, K. Atsumi, P. Ermert, *Chimia* **1999**, *53*, 291 – 294.
[10] In this experiment, excess ribose was used in order to facilitate separation of the product from the reagent in the ion-exchange chromatography.
[11] F. Heinzer, M. Soukup, A. Eschenmoser, *Helv. Chim. Acta.* **1978**, *61*, 2851 – 2874.
[12] This can either be a nucleophilic assistance by an adjacent hydroxyl group in a proton-catalyzed substitution of the NH_2 group in a non-cyclic 2-amidophosphate (formation of 2,3- and 1,2-cyclophosphates in the ribose and erythrose series), and/or a (N →O) exchange at the anomeric center through proton-catalyzed C,N dissociation →C,O recombination and subsequent hydrolytic loss of ammonia.
[13] Examples in synthetic chemistry where this strategy has used are legion. For early examples, see Ref. [16] (regioselectivity in the formation of the porphyrin skeleton in Woodward's chlorophyll synthesis) and Ref. [17] (regioselectivity of a Michael addition step in a colchicine synthesis).
[14] Note that, as the procedures described here involve separation and isolation by ion-exchange chromatography requiring large volumes, scaling up these reactions may therefore have its limits.
[15] High concentrations of substrates and reagents (up to 20% and 30%, respectively) are necessary in order for the reactions to be completed within days (instead of weeks, using lower concentrations). For the special case of ribose, the transformation to the cyclophosphates **15** and **16a** belongs to those functionalizations of the ribose molecule which select the furanose form from the sugar's pyranose/furanose equilibrium (see also Footnote 61 in Ref. [18]).
[16] a) R. B. Woodward, *Angew. Chem.* **1960**, *72*, 651 – 662; b) R. B. Woodward, *Pure Appl. Chem.* **1961**, *2*, 383 – 404.
[17] a) J. Schreiber, W. Leimgruber, M. Pesaro, P. Schudel, A. Eschenmoser, *Angew. Chem.* **1959**, *71*, 637 – 640; b) J. Schreiber, W. Leimgruber, M. Pesaro, P. Schudel, T. Threlfall A. Eschenmoser, *Helv. Chim. Acta* **1961**, *44*, 540 – 597.
[18] A. Eschenmoser, *Science* **1999**, *284*, 2118 – 2124.
[19] Further experimental details of the preparation of and spectral data for **6**, **8** + **9**, **11**, **14**, **18**, and **19** + **20** are given in the Supporting Information.

A New Gold-Catalyzed C–C Bond Formation**

A. Stephen K. Hashmi,* Lothar Schwarz, Ji-Hyun Choi, and Tanja M. Frost

While a manifold on the stoichiometric organometallic chemistry of gold is known, only a few catalytical applications have been reported.[1] As far as the *homogeneous* catalysis of organic reactions is concerned, there exist only two reactions that have reached any importance. The first forms C–X bonds (X = heteroatom) by the addition of O- or N-nucleophiles to alkenes or alkynes, as developed by Utimoto et al.[2] and Teles et al.[3] The second application forms C–C bonds by the asymmetric aldol reaction, as developed by Ito and Hayashi et al. and Togni et al.[4] We now report a new gold-catalyzed reaction that combines both C–O and C–C bond formation and allows the selective cross cycloisomerization/dimerization of terminal allenyl ketones and α,β-unsaturated ketones.

In the course of our investigation of transition metal catalyzed reactions of the readily available allenyl ketones **1**,[5] we observed that certain substrates such as **1a**, in which electron-rich substituents in the Ag^I-catalyzed Marshall reaction[6] lead to **3** as the major product, also form cycloisomers/dimers **4** as minor side products. Compound **4** is a constitutional isomer of the cycloisomer/dimer **2** obtained from the Pd^{II}-catalyzed conversion of **1** (Scheme 1).[7]



1-5	yield [%] of 2	3	4	5
a: R = 4-(TBDMSO)C_6H_4				
Pd^{II}	59	-	-	
Ag^I	-	22	14	
gold catalysis		**3**[a]	**4**[a]	**5**[b]
b: R = CH_2-4-(MeO)C_6H_4		60	31	-
c: R = CH_2-4-(TBDMSO)C_6H_4		35	5	42
d: R = CH_2CH_2-4-(TBDMSO)C_6H_4		38	6	31
e: R = 3-(MeO)C_6H_4		34	38	-
f: R = 4-(O_2N)C_6H_4		88	4	-
g: R = 2-(O_2N)C_6H_4		72	8	-
h: R = 3,4-(OCH_2O)C_6H_3		29	24	-
i: R = 4-(MeS)C_6H_4		48	51	-
k: R = CH_3		47[c]	47	-
m: R = 2,5-$(MeO)_2C_6H_3$		45	36	-

Scheme 1. Cycloisomerization, dimerization, and trimerization of **1**. a) Immediate workup after consumption of the starting material. b) Workup after several hours. c) Determined in the crude product by ^{1}H NMR.

In order to make **4** the major product, we tested Au^{III} catalysts, which combine a Pd^{II}-like d^8 system, with a silver-like metal of the copper triad. These gold catalysts prove to be extremely active and allow the reactions to be conducted under very mild conditions at room temperature or below. Because of the absence of paramagnetic species, the reactions could be easily monitored by NMR. For 1 mol% of catalyst, the required reaction times at 20 °C were, depending on the substrate, over one week for $AgNO_3$, about one hour for $[PdCl_2(MeCN)_2]$, and about one minute for $AuCl_3$! The

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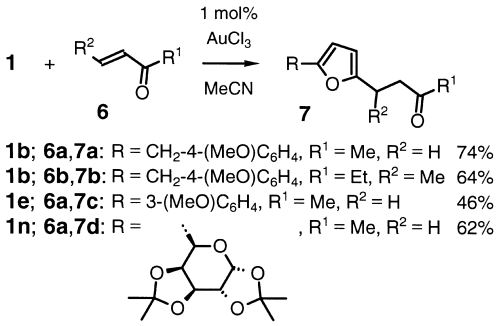
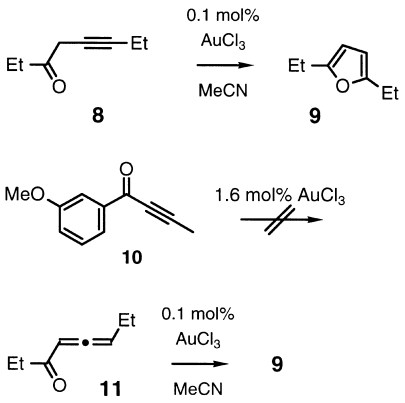
amount of Au^{III} could be reduced to 0.1 mol% with a complete consumption of **1**.

Interestingly, the initial products formed in the gold-catalyzed reactions are still **3** and **4** but the share of **4** increased. Furthermore, on a time scale of several hours, **3** and **4** reacted with each other to form **5** (a trimer of **1**) until either **3** or **4** were fully consumed. The latter observation suggested that, unlike the palladium-catalyzed conversions of **1**, in which a cross dimerization of **1** with other acceptor-substituted olefins failed, gold catalysts might allow such a cross coupling. Indeed, the addition of **1** to a solution of the catalyst and α,β-unsaturated ketones **6** did provide **7** (Scheme 2).



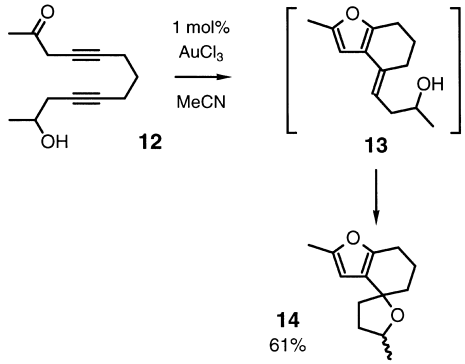
Scheme 2. Cross dimerization of **1** with Michael acceptors **6**.

These gold catalysts were also quite successful in other areas: While the reactions of propargyl ketones such as **8** failed with Ag^{I} as a catalyst and with palladium catalysts only at 100 °C,[8] the Au^{III} catalyst readily transferred **8** to **9** within minutes at room temperature in an essentially quantitative yield (Scheme 3). This saves the extra step of propargyl ketone isomerization to the corresponding allenyl ketone. On the other hand, under similar conditions the (to **1** and **8** isomeric) 1-propynyl ketones **10** did not react with the Au^{III} catalyst.



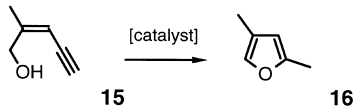
Scheme 3. Other gold-catalyzed cycloisomerizations.

Whereas for substrates like **8** or **11**, that lead to 2,5-disubstituted furans **9**, no intermolecular C–C bond formation was possible but, as shown for **12**, intramolecular C–C bonds could still form. Probably, the intermediate **13** formed and then suffered additional, nondiastereoselective C–O bond formation (no facile selection at the olefin) to provide **14** (Scheme 4).



Scheme 4. Combination of a C–C and two C–O bond forming reactions.

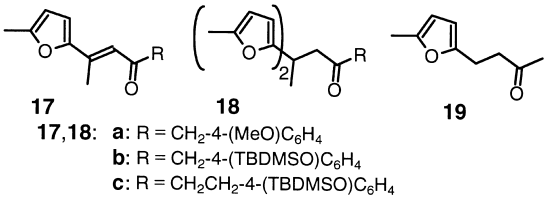
The high activity of the gold catalysts for C–O bond formation, shown already for intermolecular cases by Teles, also applies for intramolecular cases such as the transformation of **15** to **16**. While Dixneuf et al. reported that the transformation requires either 1 mol% of a Ru catalyst for 2 h treatment at 60 °C or 1 mol% of a Pd catalyst for 2 h at 100 °C,[9] and Marshall used 10 mol% of Ag catalysts for 2 h at 20 °C,[10] only 0.1 mol% of the Au catalyst for 1 h at 20 °C are sufficient (Scheme 5).



Scheme 5. Cycloisomerization of (*Z*)-enynol **15**.

Due to the high activity of the gold catalysts, we also tested whether reactions known to be catalyzed by relatively large amounts of silver catalysts are, in fact, a result of gold impurities in the silver. We found that highly pure $AgNO_3$ also showed a normal activity.[11]

For the mechanism of the reactions, the formation of **5** from **3** and **4** suggested already that two separate reactions occur, the first being the cycloisomerization of **1** to **3**, the second being the subsequent reaction of **3** with either a second molecule of **1**, **4**, or **6**. This hypothesis was further supported by the ability of $AuCl_3$ to catalyze the reaction of 2-methylfuran **3k** with **1b–d** or **6a** to form **17** and **18** or **19**, respectively (Scheme 6).



Scheme 6. Products from the cross dimerization of 2-methylfuran with allenyl ketones or methyl vinyl ketone (MVK).

Thus, for the reaction of **3** with the Michael acceptors, there remain two possibilities that both lead to the same intermediate **21**. Either the gold activates the enones, which then form the new C–C bonds by an electrophilic aromatic substitution at the 5-position of the furan to provide **21**, or a direct electrophilic attack of the gold catalyst at the furan

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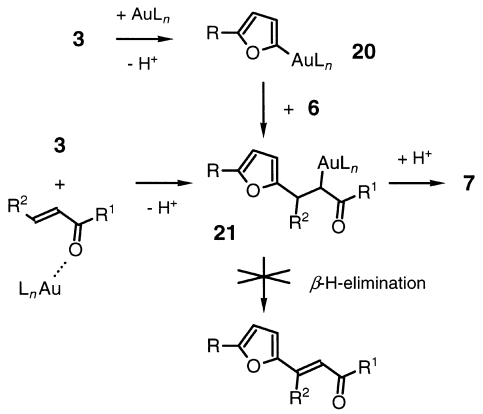
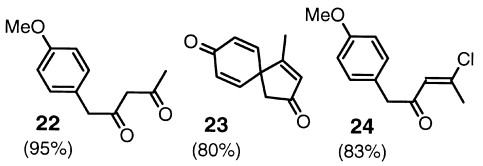
(auration)[12] forms a furyl–gold species **20**, which subsequently undergoes a 1,4-addition to the Michael system (similar to the corresponding organocuprates but generated in a protic environment; Scheme 7).



Scheme 7. Possible mechanisms for the C–C bond formation.

The gold enolate common to both routes does not undergo a β-H elimination but instead protonation by the proton set free during the electrophilic aromatic substitution step. Compound **21** strongly resembles the analogous intermediate of a palladium-catalyzed Heck reaction but, in the latter case, only in rare cases has protonation instead of β-H elimination been observed.[13] There exists one precedent in the literature that reports β-H eliminations are relatively slow with a gold catalyst.[14] As a control experiment, we mixed stoichiometric amounts of a) $AuCl_3$ and methyl vinyl ketone (MVK) or b) $AuCl_3$ and 2-methylfuran and monitored the behavior by NMR spectroscopy. For case (a) no significant change in the spectra was observed, whereas for case (b) an immediate darkening of the solution and a strong change in the spectra was observed. This change was time dependent and only broad signals were visible in the NMR spectra. While an immediate addition of MVK still provided the coupling product, a later addition did not lead to coupling (perhaps because of aggregation of the gold species, multiple aurations, or by a slow reduction of the gold).[12] We consider $AuCl_3$ only to be a precatalyst and we do not know whether the catalytically active species is Au^{III} or Au^{I} (but it should be an electrophilic gold species).[15] We occasionally observed the precipitation of gold in the form of a gold mirror but this always ocurred after the reaction was complete.

In order to prove that the Brønsted acid (H^+) set free in the electrophilic aromatic substitution was not responsible for the C–C bond formation, we treated **1b** as well as 2-methylfuran with MVK in the presence of 5% $HClO_4$. In the first case, the dione **22** was produced; in the second case, an unidentified polymeric material was formed. Compound **1b** is also a substrate that nicely proves the unique behavior of the gold catalyst: With Ag^{I} **3b** is formed; with Pd^{II}, **2b**; with H^+, **22** (Scheme 8); with Hg^{II}, **23**; with a stoichiometric amount of $FeCl_3$, **24**; with Au^{III}, **3b** and **4b**; and with Au^{III} in the presence of **6a**, the cross coupling product **7a**. So, unlike the addition of



Scheme 8. Products from the reactions of **1b** with different catalysts.

O-based nucleophiles to C–C multiple bonds, in which mercury and gold behave similarly, entirely different reactions were observed here.

Experimental Section

A solution of $AuCl_3$ in MeCN ($AuCl_3$ 30.3 mg, MeCN 970 mg) was prepared. An aliquot of this solution (267 mg; $AuCl_3$ 8.1 mg, 1 mol%) was added to **1b** (500 mg, 2.66 mmol) in MeCN (3 mL), at 20 °C. When the reaction was complete (as monitored by thin-layer chromatography (TLC)), the MeCN was removed in vacuo and the residue was purified by column chromatography (silica gel, hexane/ethyl acetate (10/1)). Compounds **3b** (298 mg, 60%) and **4b** (153 mg, 31%) were obtained. **4b**: IR (film): $\tilde{\nu}$ = 2999, 2954, 2933, 2835, 1672 cm^{-1} (C=O); ^1H NMR ($CDCl_3$, 400 MHz): δ = 2.39 (d, J = 1.1 Hz, 3H), 3.74 (s, 2H), 3.80 (s, 3H), 3.81 (s, 3H), 3.93 (s, 2H), 6.03 (d, J = 3.4 Hz, 1H), 6.60 (d, J = 3.4 Hz, 1H), 6.71 (d, J = 1.0 Hz, 1H), 6.87–6.90 (m, 4H), 7.15–7.19 (m, 4H); ^{13}C NMR ($CDCl_3$, 62.9 MHz): δ = 14.98 (q), 33.75 (t), 50.79 (t), 55.08 (q), 55.11 (q), 108.91 (d), 113.86 (d), 113.89 (d, 2C), 113.94 (d, 2C), 117.08 (d), 127.09 (s), 129.05 (s), 129.64 (d, 2C), 130.35 (d, 2C), 141.33 (s), 153.25 (s), 157.47 (s), 158.33 (s), 158.35 (s), 198.31 (s); MS (70 eV): m/z (%): 376 (14)[M^+], 255 (100); elemental analysis for $C_{24}H_{24}O_4$: calcd: C 76.57, H 6.43; found: C 76.31, H 6.50.

A 172 mg sample of a second $AlCl_3$/MeCN solution ($AuCl_3$ 30.3 mg, MeCN 967 mg; $AuCl_3$ 5.2 mg, 1 mol%) was diluted into 500 µL of MeCN containing 180 mg **6a** (2.57 mmol, 1.5 equiv). A solution of **1b** (322 mg, 1.71 mmol) in MeCN (2 mL) was then added slowly. When the reaction was complete (TLC), the MeCN was removed in vacuo and the residue was purified by column chromatography (silica gel, hexane/ethyl acetate (5/1)). Compounds **3b** (22.1 mg, 7%) and **7a** (326 mg, 74%) were obtained. **7a**: m.p.: 30–33 °C; IR (film): $\tilde{\nu}$ = 3000, 2908, 2836, 1716 cm^{-1} (C=O); ^1H NMR ($CDCl_3$, 250 MHz): δ = 2.13 (s, 3H), 2.70–2.77 (m, 2H), 2.83–2.90 (m, 2H), 3.79 (s, 3H), 3.85 (s, 2H), 5.83 (d, J = 3.0 Hz, 1H), 5.87 (d, J = 3.0 Hz, 1H), 6.82–6.87 (m, 2H), 7.11–7.15 (m, 2H); ^{13}C NMR ($CDCl_3$, 62.9 MHz): δ = 22.19 (t), 29.73 (q), 33.48 (t), 41.64 (t), 55.09 (q), 105.60 (d), 106.40 (d), 113.70 (d, 2C), 129.47 (d, 2C), 130.20 (s), 153.12 (s), 153.39 (s), 158.06 (s), 207.25 (s); MS (70 eV): m/z (%): 258 (100)[M^+]; elemental analysis for $C_{16}H_{18}O_3$: calcd: C 74.40, H 7.02; found: C 74.12, H 6.94.

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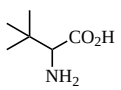
[15] Au^I, in the form of [AuCl(tetrahydrothiophene)], converted **1b** to **3b** only and no dimer **4b** was observed.

α-Trialkylsilyl-Substituted α-Amino Acids**

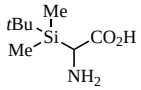
Carsten Bolm,* Andrey Kasyan, Karlheinz Drauz, Kurt Günther, and Gerhard Raabe

Dedicated to Professor Franz Effenberger on the occasion of his 70th birthday

As building blocks of peptides and proteins, α-amino acids are widespread throughout nature.[1] Various methods have been developed to gain access to them,[2] and among these catalytic processes appear particularly attractive.[2d] Among the nonproteinogenic amino acids *tert*-leucine (**1**) has emerged as an outstanding representative.[3] Bearing a hydrophobic and sterically demanding *tert*-butyl substituent, it has proved to be a valuable building block for pharmacologically effective peptide analogues[4] and has been frequently employed in syntheses of chiral auxiliaries.[5, 6] Here, we report the first synthesis of *tert*-leucine-analogous α-trialkylsilyl-substituted α-amino acids **2** and their corresponding, amino- and carboxyl-protected derivatives **4**.[7]

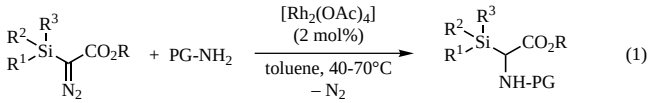


1



2

For the synthesis of **2**, fully protected α-trialkylsilyl-α-aminoacetates **4** play a pivotal role. These are prepared starting from α-trialkylsilyl-α-diazoacetates **3**,[8] which in turn are accessible through reaction of the corresponding α-diazoacetates with trialkylsilyltriflates in the presence of tertiary amine bases.[9, 10] Rhodium-catalyzed cleavage of nitrogen[11, 12] and intermolecular carbenoid-type N,H-insertion[12, 13] leads to the protected α-trialkylsilyl-α-aminoacetates **4** [Eq. (1); PG = protecting group]. The yields in these



(1)

3 **4**

insertion reactions are good (up to 86 %; Table 1). An X-ray crystal structure analysis of ester *rac*-**4a** confirmed its constitution.[14]

Table 1. Synthesis of protected α-trialkylsilyl-α-aminoacetates **4**.

R	R^1/R^2	R^3	PG[a]	Product	Yield [%]
Et	Me	*t*Bu	Tos	**4a**[b]	58
Et	Me	*t*Bu	Boc	**4b**	72
Et	Me	*t*Bu	Z	**4c**	83
Et	Me	Me	Boc	**4d**	65
Et	Me	Me	Z	**4e**	69
Et	Et	Et	Boc	**4f**	77
Et	Et	Et	Z	**4g**	86
Bn	Me	*t*Bu	Boc	**4h**	47
Bn	Me	*t*Bu	Z	**4i**	53

[a] Tos = tosyl = *p*-toluenesulfonyl, Boc = *tert*-butoxycarbonyl, Z = benzyloxycarbonyl. [b] From a reaction sequence as described in the Experimental Section.

The enantiomers of **4a** were separated by means of preparative HPLC using a chiral stationary phase.[15] Enantiopure **4a** is configurationally stable even upon prolonged storing. The absolute configuration of the later-eluted (+)-enantiomer of **4a** was determined to be (*S*) by a second X-ray crystal structure analysis. Figure 1 shows the molecular structure of (*S*)-**4a** in the crystal.[14]

The Si–C11 bond (1.919(5) Å) in (*S*)-**4a** is significantly longer than those between the silicon atom and C13 (1.904(6) Å), C12 (1.862(5) Å), and C17 (1.865(4) Å); the last two bond lengths fall within the range that is considered average for a bond between a four-coordinate Si atom and an

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