Working with Hazardous Chemicals

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*September 2014: The paragraphs above replace the section “Handling and Disposal of Hazardous Chemicals” in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*
Preparation of 1-Benzyl-2-methyl-3-(ethoxycarbonyl)-4-(2-phenylethyl)-1H-pyrrole from 4-Phenyl-1,2-butadiene

A. 4-Phenyl-1,2-butadiene (1). A 3-necked 250-mL round-bottomed flask containing a magnetic stir bar (3 cm length x 1.5 cm diameter) is equipped with a reflux condenser and two rubber septa (Note 1). The top of the condenser is fitted with a stopcock connected to a vacuum line with an argon flow. Magnesium turnings (7.2 g, 0.3 mol, 1.2 equiv) are added and the apparatus is flame-dried under vacuum. After cooling to room temperature under an argon purge, the flask is charged with diethyl ether (100 mL) and equipped with a thermometer. 1,2-Dibromoethane (0.5 mL) (Note 2) is added to initiate the reaction with a slight increase of the internal temperature (2–3 °C). Benzyl bromide (30 mL, 0.25 mol) (Note 2) is injected into the mixture with a syringe pump at a rate so as to maintain a

Caution! Ethyl α-diazoacetoacetate is a potentially explosive material. Transformations involving this compound should be performed behind a blast shield in a well-ventilated fume hood.

Submitted by Shengjun Ni, Can Zhu, Jie Chen and Shengming Ma. *1
Check by Mélanie Charpenay and Kay Brummond.

1. Procedure

A. 4-Phenyl-1,2-butadiene (1). A 3-necked 250-mL round-bottomed flask containing a magnetic stir bar (3 cm length x 1.5 cm diameter) is equipped with a reflux condenser and two rubber septa (Note 1). The top of the condenser is fitted with a stopcock connected to a vacuum line with an argon flow. Magnesium turnings (7.2 g, 0.3 mol, 1.2 equiv) are added and the apparatus is flame-dried under vacuum. After cooling to room temperature under an argon purge, the flask is charged with diethyl ether (100 mL) and equipped with a thermometer. 1,2-Dibromoethane (0.5 mL) (Note 2) is added to initiate the reaction with a slight increase of the internal temperature (2–3 °C). Benzyl bromide (30 mL, 0.25 mol) (Note 2) is injected into the mixture with a syringe pump at a rate so as to maintain a
gentle reflux (15 mL/h). After the addition, the mixture is heated under reflux with an oil bath (oil bath temperature, 55 °C) for another 2 h. Another 3-necked 250-mL round-bottomed flask equipped with a magnetic stir bar (3 cm length x 1.5 cm diameter) (Note 1) is fitted with two rubber stoppers and a 250-mL pressure equalizing addition funnel. The flask and funnel are flame-dried under vacuum. After cooling to room temperature under an argon purge, propargyl bromide (33.4 mL of an 80 wt. % solution in toluene, 0.3 mol, 1.2 equiv) (Note 2) and diethyl ether (50 mL) (Note 2) are added and the flask is fitted with a thermometer. The mixture is cooled to 0 °C (internal temperature) with an ice-salt bath. The prepared Grignard reagent is transferred to the addition funnel using an 18-gauge cannula and added to the propargyl bromide over 3 h. After the addition, the resulting mixture is stirred for an additional 2 h at 0 °C (Note 3). Then the mixture is poured into a 1-L Erlenmeyer flask containing a cold saturated aqueous solution of NH₄Cl (200 mL). The mixture is transferred to a 500 mL separatory funnel, the phases are separated, and the aqueous phase is back-extracted with diethyl ether (100 mL x 2) (Note 2). The combined organic phases are washed with brine (100 mL), dried over anhydrous Na₂SO₄, and filtered. The solvent is removed by rotary evaporation (20 mmHg, 30 °C water bath). Purification is performed by vacuum distillation (70–80 °C/23 mmHg, fractionating column: 2 cm diameter x 15 cm length) to afford 4-phenyl-1,2-butadiene (1) (18.6 g, 57% by the checker; 24.9 g, 77% by the submitter, see discussion) as a colorless liquid (Note 4).

B. 1-Acetyl-2-phenethylidene cyclopropanecarboxylic acid ethyl ester (2). A 3-necked 250-mL round-bottomed flask is equipped with a reflux condenser, the top of which is fitted with a stopcock connected to a vacuum line with an argon flow. The other two necks are fitted with rubber septa. After evacuating and backfilling with argon the flask is charged with a magnetic stir bar (3 cm length x 1.5 cm diameter), Rh₂(OAc)₄ (180 mg, 0.4 mmol, 0.008 equiv) (Note 5), 4-phenyl-1,2-butadiene (1) (19.5 g, 150 mmol, 3.00 equiv) (Note 6) and CH₂Cl₂ (40 mL) (Note 2) under argon. The mixture is heated to reflux using an oil bath (55 °C, external temperature) and after 5 min at reflux, a solution of ethyl α-diazoacetoacetate (7.8 g, 50 mmol, 1 equiv) in CH₂Cl₂ (10 mL) is added using a syringe pump (10 mL/h). Upon completion of the addition, an additional quantity of Rh₂(OAc)₄ (40 mg, 0.1 mmol, 0.002 equiv) (Note 7) is added and the resulting mixture is refluxed for 2 h; the progress of the reaction is monitored by TLC (Note 3). Upon completion, the reaction is cooled to
room temperature and concentrated by rotary evaporation (20 mmHg, 35 °C water bath). The residue is purified by column chromatography (column ø = 80 mm with 300 g of silica gel (230-400 mesh); ~150 mL fractions; n-Hexane (1.5 L) (Note 2) is used to recover 4-phenyl-1,2-butadiene 1 (fractions 5-12, 7.64 g, 39%); then a mixture of 3:1 n-hexane:ethyl acetate (Note 2) (2 L) is used to elute 1-acetyl-2-phenethylidene-cyclopropanecarboxylic acid ethyl ester 2 (fractions 20-25, 15.1 g) (Note 8).

C. 1-Benzyl-2-methyl-3-(ethoxycarbonyl)-4-(2-phenylethyl)-1H-pyrrole (3). To a 100-mL round-bottomed flask is added, in succession, a magnetic stir bar (3 cm length x 1.5 cm diameter) (Note 1), crude product 2 (15.0 g), CH₃CN (15 mL, Note 2), benzylamine (9.8 mL, 90 mmol) (Note 2) and CH₃CN (15 mL). The flask is equipped with a reflux condenser and the top of the condenser left open to the air. After being heated with an oil bath (80 °C) for 19 h the reaction is judged complete as evidenced by TLC (Note 3). The resulting mixture is concentrated by rotary evaporation (20 mmHg, 35 °C water bath) and the residue is purified by column chromatography (column ø = 80 mm, 300 g of silica gel, 230-400 mesh). A mixture of petroleum ether (8.0 L) (Note 2) and ethyl acetate (0.2 L) (Note 2) are used as eluent, collecting ~150 mL fractions. Concentration of fractions 21-50 by rotary evaporation (20 mmHg, 40 °C water bath) affords 1-benzyl-2-methyl-3-(ethoxycarbonyl)-4-(2-phenylethyl)-1H-pyrrole 3 (9.05 g, 52% combined yield for steps B and C) as a light yellow solid (Notes 9 and 10).

2. Notes

1. All glassware was thoroughly washed and dried in an oven (150 °C). Magnetic stir bars were washed with acetone and dried.

2. The submitters prepared their own magnesium turnings from magnesium ingot (>99%) purchased from Sinopharm Chemical Reagent Co., Ltd and obtained a higher yield for the reaction (77% vs 57%). The checkers used magnesium turnings purchased from Sigma Aldrich. The submitters used anhydrous diethyl ether (≥99%) purchased from Shanghai Experimental Reagent Co., Ltd and distilled over sodium wire with diphenyl ketone as the indicator and the checkers used column-dried diethyl ether. The submitters used dichloromethane distilled from calcium hydride and the checkers used column-dried dichloromethane. Benzyl bromide (≥98%) was purchased from Sigma Aldrich and distilled from MgSO₄ before use. The
submitters purchased propargyl bromide (≥98%) from Shanghai Darui Finechemical Co., Ltd and distilled before use. The checkers purchased an 80 wt. % solution of propargyl bromide in toluene (Sigma-Aldrich) and used as received. 1,2-Dibromoethane (≥98%) was purchased from Sigma-Aldrich, petroleum ether from Fisher Scientific, and all the other chemicals from Sigma Aldrich. All chemicals were used as received unless specified above. Ethyl diazoacetoacetate was prepared using an Organic Syntheses method.2

3. TLC analysis: Step A: Rf of compound 1 = 0.68 (eluent: petroleum ether); Step B: Rf of ethyl α-diazoacetoacetate = 0.39 (eluent: petroleum ether/EtOAc (10/1)) and visualized using UV (254 nm); Rf of compound 2 = 0.42 (eluent: petroleum ether/EtOAc (10/1)) and visualized with KMnO4. Step C: Rf of compound 3 = 0.48 (eluent: petroleum ether/EtOAc (10/1)) and visualized using UV (254 nm) and an aqueous solution of KMnO4.

4. 4-Phenyl-1,2-butadiene6 was colorless after distillation under reduced pressure, but turned yellow after a few hours at room temperature. GC purity: 94.6% (conditions: Rtx-5MS column (30×0.25×0.25); oven: 50 °C, then 10 °C/min, 270 °C for 5 min; injector: 230 °C; EI, interphase temperature: 280 °C; split: 42:1; He: 3.0 mL/min). 1H NMR (400 MHz, CDCl3) δ: 3.36–3.40 (m, 2 H), 4.72–4.75 (m, 2 H), 5.30 (quint, J = 7.0 Hz, 1 H), 7.21-7.34 (m, 5 H); 13C NMR (100 MHz, CDCl3) δ: 35.3, 75.2, 89.7, 126.4, 128.5, 128.6, 140.4, 209.1; IR (neat) ν 3085, 3062, 3027, 2978, 2909, 2859, 1936, 1686, 1603, 1519, 1496, 1427, 1336, 1271, 1180, 1085, 1030, 968 cm⁻¹; MS (EI) m/z 131 (89, M+H⁺), 103 (100), 91 (98), 77 (88), 65 (80).

5. The submitters prepared rhodium(II) acetate dimer (Rh2(OAc)4) according to the procedure in Inorganic Synthesis.3 The checkers purchased Rh2(OAc)4 from Strem Chemicals.

6. The submitters reported an increase in the yield of this reaction with increasing quantities of 4-phenyl-1,2-butadiene: 2.0 equiv gave 41% yield; 3.0 equiv gave 53% yield, and 4 equiv gave 60% yield of 2. After the reaction, excess 4-phenyl-1,2-butadiene was recovered by column chromatography.

7. Rh2(OAc)4 should be added in two portions because ethyl α-diazoacetoacetate did not fully react when the same quantity of Rh2(OAc)4 was added in one portion.

8. The crude 1-acetyl-2-phenethylidenecyclopropanecarboxylic acid ethyl ester 2 (a mixture of Z/E isomers,9 ratio = 1.7:1) was used directly in the next step without further purification. A portion of this crude material (60.8 mg) was purified by chromatographic separation on silica gel with an
eluent of n-hexane:ethyl acetate = 30:1 for characterization data. $^1$H NMR (300 MHz, CDCl$_3$) $\delta$: 1.23–1.28 (m, 3 H), 2.07–2.32 (m, 2 H), 2.32–2.35 (m, 3 H), 3.50–3.56 (m, 2 H), 4.15–4.23 (m, 2 H), 6.01 (t, $J$ = 7.2 Hz, 0.34 H, =CH), 6.05–6.12 (m, 0.66 H, =CH), 7.13–7.31 (m, 5 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 14.2 (2 C), 17.7, 19.0, 28.5, 28.7, 37.5, 37.9, 39.9 (2 C), 61.6 (2 C), 118.6 (2 C), 125.1, 125.2, 126.5 (2 C), 128.6 (2 C), 128.7 (2 C), 139.3, 139.4, 169.0, 169.1, 200.4, 200.8; IR (neat) ν 1686, 1496, 1427, 1356, 1273, 1235, 1207, 1180, 1129, 1084, 1030 cm$^{-1}$; MS m/z 259 (M+H$^+$, 100), 258 (39), 213 (13), 212 (7); HRMS calcd. for C$_{16}$H$_{19}$O$_3$: 259.1334. Found: 259.1332.

9. 1-Benzyl-2-methyl-3-(ethoxycarbonyl)-4-(2-phenylethyl)-1H-pyrrole (3): mp = 42 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 1.37 (t, $J$ = 7.1 Hz, 3 H), 2.44 (s, 3 H), 2.88–2.92 (m, 2 H), 2.99–3.03 (m, 2 H), 4.32 (q, $J$ = 7.1 Hz, 2 H), 4.97 (s, 2 H), 6.30 (s, 1 H), 6.96 (d, $J$ = 6.7 Hz, 2 H), 7.16–7.34 (m, 8 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 11.8, 14.7, 29.1, 37.2, 50.4, 59.3, 111.3, 119.7, 124.9, 125.7, 126.5, 127.7, 128.2, 128.7, 128.9, 136.6, 137.3, 142.8, 166.2; MS (EI) m/z 348 (M+H$^+$, 100), 347 (4), 302 (2), 256 (3); IR (neat) ν 1950, 1686, 1642, 1564, 1506, 1496, 1445, 1428, 1355, 1339, 1273, 1235, 1180, 1129, 1084, 1030 cm$^{-1}$; HRMS (EI) calcd for C$_{23}$H$_{26}$NO$_2$ (M$^+$): 348.1964; Found: 348.1954; Anal. calcd. for C$_{23}$H$_{25}$NO$_2$: C, 79.51; H, 7.25; N, 4.03; Found: C, 79.67; H, 7.15; N, 3.89.

10. This compound was recrystallized from petroleum ether and EtOAc for melting point determination. For recrystallization, 3 (0.53 g) was dissolved in EtOAc (1 mL) with heating. Then petroleum ether (20 mL) was added. The solution was sealed and kept at −8 °C for one day. The crystals (65 mg, 12%) were collected via filtration and 465 mg (88%) was recovered by evaporation of the mother liquor.

**Handling and Disposal of Hazardous Chemicals**

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### 3. Discussion

Pyrroles are one of the most prevalent heterocyclic compounds. In recent years, polysubstituted pyrroles with various substituents have been prepared.⁴ However, there are very limited reports on syntheses of 2,3,4-trisubstituted pyrroles.⁵ In this contribution, we have demonstrated an intermolecular cyclization reaction of alkylidenecyclopropyl ketones and amines which provides an efficient route to 2,3,4-trisubstituted pyrroles.

4-Phenyl-1,2-butadiene was first prepared by the group of Hirao et al. by first generating an organovanadium compound from reaction of benzylmagnesium bromide with VCl₃, then reacting the resulting species with propargyl bromide to afford the allene in 31% yield with 4% of 4-phenyl-1-butyne as a byproduct.⁶ Other methods of generating alkyl- and aryl-substituted allenes have been mediated by CuX.⁷ However, we found 4-phenyl-1,2-butadiene could be prepared without CuX or any additional metal as catalyst. New glassware was used to ensure no residual Cu(I) was present as a contaminant. Initial efforts using this procedure resulted in the formation of >10% of 4-phenyl-1-butyne.⁷ However, its formation was diminished by slow addition of the Grignard reagent into propargyl bromide. The preparation of benzyl magnesium bromide is very important to ensure a higher yield. The checkers did not see the formation of 4-phenyl-1-butyne but instead observed a 16% recovery of propargyl bromide and an 11% yield of 1,2-diphenylethane as estimated by crude NMR, due to the less efficient formation of the benzyl magnesium bromide forming the benzyl bromide-homocoupling product 1,2-diphenylethane leading to the recovery of the propargyl bromide in the subsequent step.

We have prepared various 2,3,4-trisubstituted pyrroles via reaction of alkylidenecyclopropyl ketones with amines (Table 1).⁸ In step B, the alkylidenecyclopropylketone products are contaminated with some minor by-products, but the crude product obtained by simple filtration through a column of silica gel can be used directly for the next step. In the multi-gram
scale reaction of step C, moisture is easily controlled; thus, the reaction can be conducted without MgSO₄.

**Table 1. Intermolecular Cyclization of Alkylidenecyclopropyl Ketones with Amines Affording 2,3,4-Trisubstituted Pyrroles**

![Chemical structure](image)

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<th>R³</th>
<th>R⁴</th>
<th>yield (%)</th>
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Two possible mechanistic pathways can be envisioned to afford the corresponding pyrroles. One pathway starts from nucleophilic attack of the amine at the less sterically hindered carbon atom of the 3-membered ring to afford intermediate 4 (path a). The nucleophilic nitrogen then reacts with the carbonyl group leading to 3-alkylidene-5-hydroxy tetrahydropyrrole intermediate 5. Subsequent dehydration and aromatization generates product 3 via the intermediacy of 6. An alternative pathway starts with the intermolecular condensation of 1 and 2 to afford the cyclopropylimine intermediate 7 (path b). Cloke-type rearrangement of intermediate 7 readily leads to ring expansion via the subsequent nucleophilic attack of the nitrogen atom at the less sterically hindered carbon atom in the cyclopropane ring, which causes the distal cleavage to form 6. Subsequent aromatization affords product 3.
Scheme 1. Proposed reaction mechanism

1. State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 345 Lingling Road, Shanghai, China. E-mail: masm@sioc.ac.cn. We thank the National Natural Science Foundation of China (21232006) and the National Basic Research Program of China (2009CB825300) for financial support.


Appendix

**Chemical Abstracts Nomenclature; (Registry Number)**

Magnesium; (7439-95-4)
Benzene, (bromomethyl)-; (100-39-0)
1-Propyne, 3-bromo-; (106-96-7)
Magnesium, bromo(phenylmethyl)-; (1589-82-8)
Benzenemethanamine; (100-46-9)
Butanoic acid, 2-diazo-3-oxo-, ethyl ester; (2009-97-4)
Rhodium, tetrakis[μ-(acetato-κO:κO')]di-, (Rh-Rh); (15956-28-2)
Ethane, 1,2-dibromo-; (106-93-4)
Prof. Shengming Ma was born in 1965 in Zhejiang, China. He graduated from Hangzhou University (1986) and received his Ph.D. degree from Shanghai Institute of Organic Chemistry (1990). He became an assistant professor of SIOC in 1991. After his postdoctoral appointments at ETH with Prof. Venanzi and Purdue University with Prof. Negishi from 1992–1997, he joined the faculty of SIOC in 1997. From February 2003 to September 2007, he was jointly appointed by SIOC and Zhejiang University. He works for East China Normal University and SIOC from October 2007.

Shengjun Ni was born in Jining, China. He received his B.S. degree in applied chemistry from China University of Petroleum (CUP) in 2007. He is now pursuing his M.S. degree in Shanghai Institute of Organic Chemistry (SIOC), Chinese Academy of Sciences under the supervision of Prof. Shengming Ma. His research interest is transition metal catalyst in modern organic synthesis.

Can Zhu was born in Nantong, China. He received his B.Sc. in chemistry from University of Science and Technology of China (USTC) in 2009. He is currently pursuing a Ph.D. in Shanghai Institute of Organic Chemistry (SIOC) under the supervision of Prof. Shengming Ma, working on the application of transition metal catalysis in modern organic synthesis.
Jie Chen was born in Nanyang, China. She received her B.Sc. in chemistry from Henan University in 2005. In 2010, she obtained her Ph.D. under the supervision of Prof. Shengming Ma from Shanghai Institute of Organic Chemistry (SIOC), Chinese Academy of Sciences. In 2011, she joined the laboratory of Prof. Joseph M. Ready as a postdoctoral fellow at the University of Texas Southwestern Medical Center (UTSW).

Mélanie Charpenay was born in 1986 in Voiron, France. She studied organic chemistry at the European Engineering School of Chemistry, Polymers and Materials Science in Strasbourg and graduated in 2009. The same year, she obtained her Masters Degree at the University of Strasbourg. Then she carried out Ph.D. studies at the same university under the supervision of Dr. Jean Suffert, focusing on the development of new synthetic methodologies to reach highly strained polycyclic structures such as fenestradienes and cyclooctatrienes using palladium catalyzed cascade reactions. She is currently working as a postdoctoral fellow at the University of Pittsburgh with Professor Kay Brummond and group on the synthesis of biologically relevant fluorescent probes.
propargyl bromide solution in toluene (1D 1H)

Parameters
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  - Carrier Frequency: 300.23184 MHz
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- Temperature: -928.2401 K
- Solvent: CDCl3

Molecular Weight: 182.27
Parameters

compound 1 proton (1D 1H)

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- Spectral Width : 8223.685 Hz
- Carrier Frequency : 400.13248 MHz
- Nucleus : 1H

Number of scans : 32

Type : 1D
Spectro : BRUKER 400 MHz
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Date : Sat Mar 16 22:54:43 EDT 2013
Temperature : 297.5 K
Solvent : CDCl3
compound 2 crude (1D 1H) CDCl3 400MHz

Parameters
mc4lf2 10 (1D 1H)

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- Size: 65536 points complex
- Spectral Width: 8223.685 Hz
- Carrier Frequency: 400.13248 MHz
- Nucleus: 1H

Number of scans: 16

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3.08-2.82 ppm
compound 3 carbon (1D 13C) CDCl₃ 400MHz

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- Spectro: BRUKER 400 MHz
- Probe: 5 mm PABBO BB-1H/D Z-GRD Z101
- Date: Tue Mar 19 22:01:05 EDT 2013
- Temperature: 297.9 K
- Solvent: CDCl₃

128.8-125 ppm