

Benzil (22) and Vinyl Phenyl Ketone (24). By use of the same procedure, the hydrolyses of 21 (161 mg, 0.4 mmol) and 23 (114 mg, 0.5 mmol) were carried out to give 22: [45 mg (53% yield); mp 92–93 °C (lit.¹⁴ mp 93–95 °C)] and 24: 42 mg (64% yield); bp 98–100 °C (10 mmHg) [lit.¹⁵ bp 114–115 °C (17 mmHg)].

Registry No. 1a, 827-36-1; 1b, 5097-99-4; 1c, 31466-31-6; 1d, 15190-10-0; 1e, 15190-67-7; 2a, 85356-09-8; 2b, 85356-10-1; 2c, 85356-11-2; 2d, 85356-12-3; 3a, 85356-13-4; 4a, 85356-14-5; 5a, 85356-15-6; 6, 495-71-6; 7, 6263-83-8; 8, 6268-58-2; 9, 66901-95-9; 10a, 15190-05-3; 11a, 15190-08-6; 12a, 15190-02-0; 13a, 85356-16-7; 14a, 85356-17-8; 15a, 85356-18-9; 16a, 85356-19-0; 17, 15982-64-6; 18, 24314-35-0; 19, 13669-05-1; 20, 37709-57-2; 21, 85356-20-3; 22, 134-81-6; 23, 85356-21-4; 24, 768-03-6; TsOCH₂CH₂OTs, 6315-52-2; 1,2-dibromoethane, 106-93-4; 1,3-dibromopropane, 109-64-8; 1,6-dibromohexane, 629-03-8; 1,10-dibromodecane, 4101-68-2.

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Cross-Coupling Reaction of 1,3-Butadien-2-ylmagnesium Chloride with Alkyl or Aryl Halides by Lithium Chloride-Cupric Chloride (Li₂CuCl₄), a Superior Catalyst

Sadaaki Nunomoto*

Faculty of Chemical Engineering, Toyama Technical College, Hongo, Toyama 930, Japan

Yuhsuke Kawakami* and Yuya Yamashita

Department of Synthetic Chemistry, Faculty of Engineering, Nagoya University, Chikusa, Nagoya 464, Japan

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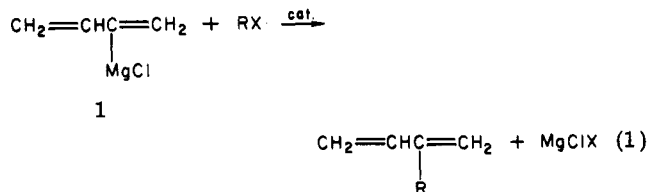
Cross-coupling reactions of Grignard reagents have been reported with alkyl, allyl, alkenyl, and aryl halides in the presence of various kinds of transition-metal catalysts.¹⁻⁸ The authors also reported that copper(I) iodide (CuI), and tetrakis(triphenylphosphine)palladium(0) (Pd(PPh₃)₄) were effective catalysts in the cross-coupling reactions of 1,3-butadien-2-ylmagnesium chloride (1) with alkyl and aryl iodides, respectively.⁹

In the course of our studies in exploring more efficient and selective catalyst systems, Li₂CuCl₄, initially reported by Tamura and Kochi,² was found to be a very effective and selective catalyst for the coupling reactions of 1 with primary alkyl iodides or bromides. In the cross-coupling reactions of Grignard reagent, Li₂CuCl₄ generally appears to be quite a good catalyst. Fouquet¹⁰ and Chapman¹¹

improved the yields by the use of tosylate as a leaving group. Selective coupling of ω-bromo acid was also reported by Baer.¹²

In this note, we report the syntheses of 2-substituted 1,3-butadienes and selective cross-coupling reactions of mixed halides taking advantage of the high reactivity of primary alkyl iodide and bromide.

Cross-Coupling Reactions of 1 with Octyl Halides. The results of the cross-coupling reactions of Grignard reagents with octyl halides are shown in Table I.



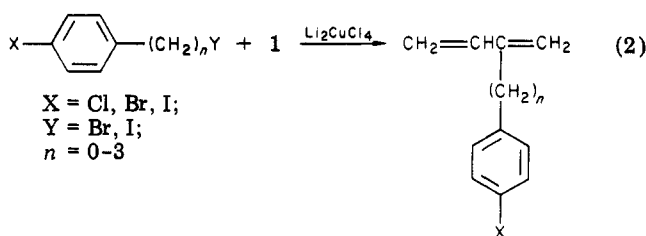
The reactions with CuI as catalyst gave only reasonable yields. Even with the iodide (no. 1, 2) considerably large amounts of the catalyst were required. On the contrary, good to excellent yields could be obtained even with the bromide by employing only small amounts of Li₂CuCl₄ as catalyst (no. 6, 7).

Although a little longer reaction time is required, the Li₂CuCl₄ system has several advantages over the CuI system: namely, moderate reaction temperature, smaller amounts of catalyst, higher yield of the product, and easiness of the separation of the product.

The order of reactivity of the halides was I > Br >> Cl, and secondary halides showed much lower reactivity than primary halides.

Cross-Coupling Reactions of 1 with Aryl Halides. The results of the cross-coupling with aryl halides are shown in Table II. Despite the relatively lower reactivities of aryl halides, reasonable yields of the cross-coupled product could still be obtained from aryl iodide substrate (no. 17). The bromide showed considerably lower reactivity than iodides.

Taking advantage of the differences in reactivity, the selective cross-coupling reactions of the aromatic dihalides were investigated (no. 18–22).



In the cross-coupling reactions of 1 with halides having both aromatic and aliphatic halogens, yields as high as 87% of the selectively coupled product, namely, 2-halogenoaromatic-substituted butadienes, could be obtained. It was interesting to note that the aromatic halogen was not affected at all in the reaction.

However, with Pd(PPh₃)₄ as catalyst, the aromatic iodide preferentially reacted (no. 23).

Cross-Coupling Reactions of 1 with Functional Halides. The cross-coupling reactions of 1 with halides containing other functional groups were also investigated, and the results are given in Table III. The cross-coupling

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Table I. Cross-Coupling Reactions of Grignard Reagents with Octyl Halides in THF^a

no.	halides	RMgX	catalyst (mol %)	temp, °C	time, h	cross-coupling product yield, %
1 ^b	<i>n</i> -C ₈ H ₁₇ Br	1	CuI (20)	-30-0	2	5
2 ^b	<i>n</i> -C ₈ H ₁₇ I	1	CuI (20)	-30-0	2	60
3	<i>n</i> -C ₈ H ₁₇ Cl	1	Li ₂ CuCl ₄ (5)	65	3	5
4	<i>n</i> -C ₈ H ₁₇ Br	1	Li ₂ CuCl ₄ (1)	20	16	25
5	<i>n</i> -C ₈ H ₁₇ Br	1	Li ₂ CuCl ₄ (5)	20	16	82
6	<i>n</i> -C ₈ H ₁₇ Br	1	Li ₂ CuCl ₄ (2)	65	2	80
7	<i>n</i> -C ₈ H ₁₇ I	1	Li ₂ CuCl ₄ (2)	20	16	95
8	<i>n</i> -C ₆ H ₁₃ CH(CH ₃)Br	1	Li ₂ CuCl ₄ (5)	65	3	5
9	<i>n</i> -C ₆ H ₁₃ CH(CH ₃)I	1	Li ₂ CuCl ₄ (5)	20	16	45
10	<i>n</i> -C ₈ H ₁₇ Br	<i>i</i> -PrMgBr	Li ₂ CuCl ₄ (3)	20	16	24
11	<i>n</i> -C ₈ H ₁₇ I	<i>i</i> -PrMgBr	Li ₂ CuCl ₄ (3)	65	2	78 ^c
12	<i>n</i> -C ₈ H ₁₇ Br	C ₆ H ₅ MgBr	Li ₂ CuCl ₄ (3)	20	16	76
13	<i>n</i> -C ₈ H ₁₇ Br	CH ₂ =CHMgBr	Li ₂ CuCl ₄ (3)	20	3	24
14	<i>n</i> -C ₈ H ₁₇ I	CH ₂ =CHMgBr	Li ₂ CuCl ₄ (3)	20	16	36 ^c

^a [RMgX]₀ = 1 mol/L, [RMgX]:[RX] = 1:1 molar ratio. ^b After the completion of the addition of 1 at -30 °C, stirring was continued for the prescribed time interval at 0 °C. ^c 10-20% of homo-coupling product C₁₆H₃₄ was observed.

Table II. Preparation of Butadiene Derivatives from 1 and Aryl Halides in THF by the Use of Li₂CuCl₄^a

no.	ArX or RX	catalyst amount, mol %	temp, °C	time, h	cross-coupling product	
					yield, %	bp, °C/mmHg
15 ^{b,c}	C ₆ H ₅ I	3	reflux temp	1	75	62-65/20
16	C ₆ H ₅ Br	3	65	2	5	
17	C ₆ H ₅ I	5	20	20	45	
18	<i>p</i> -BrC ₆ H ₄ I	5	20	20	40	80-82/2
19	<i>p</i> -ClC ₆ H ₄ CH ₂ Br	3	20	16	70	81-86/2
20	<i>p</i> -BrC ₆ H ₄ CH ₂ Br	3	20	5	70	93-98/2
21	<i>p</i> -BrC ₆ H ₄ CH ₂ CH ₂ I	3	20	16	87	102-103/2
22	<i>p</i> -IC ₆ H ₄ CH ₂ CH ₂ CH ₂ I	3	20	16	85	120-125/2
23 ^{b,c}	ICH ₂ CH ₂ CH ₂ C ₆ H ₄ <i>i</i> -p	3	reflux temp	1	60 ^d	

^a [RMgX]₀ = 1 mol/L, [RMgX]:[RX] = 1:1 molar ratio. ^b In this run, Pd(PPh₃)₄ was used as catalyst instead of Li₂CuCl₄. ^c In THF-C₆H₆ (100 mL + 50 mL). ^d Determined by GLC.

Table III. Preparation of Butadiene Derivative from 1 and Functional Halogen Compounds^a

no.	YRX	catalyst (mol %)	temp, °C	time, h	cross-coupling product	
					yield, %	bp, °C/mmHg
24 ^b	CH ₃ OCOCH ₂ CH ₂ I	CuI (20)	-30-0	2	polymerized	
25 ^b	C ₂ H ₅ OCO(CH ₂) ₂ Br	CuI (20)	-30-0	2	20	
26 ^b	CH ₃ OCO(CH ₂) ₃ I	CuI (20)	-30-0	2	65	
27 ^b	CH ₃ OCO(CH ₂) ₄ I	CuI (20)	-30-0	2	68	
28	ClCH ₂ CH ₂ CH ₂ Br	Li ₂ CuCl ₄ (5)	20	16	40	
29	BrCH ₂ CH ₂ CH ₂ CH ₂ Br	Li ₂ CuCl ₄ (5)	20	16	40	
30 ^c	C ₆ H ₅ COOCH ₂ CH ₂ I	Li ₂ CuCl ₄ (5)	0-20	16	86	
31 ^d	HOCH ₂ CH ₂ I	Li ₂ CuCl ₄ (3)	65	2	75	
32 ^c	CH ₃ OCO(CH ₂) ₄ I	Li ₂ CuCl ₄ (3)	0-20	16	80	
33 ^c	NCCH ₂ CH ₂ CH ₂ Br	Li ₂ CuCl ₄ (3)	0-20	16	45	
34	C ₆ H ₅ OCH ₂ CH ₂ Br	Li ₂ CuCl ₄ (3)	65	3	78	

^a [RMgX]₀ = 1 mol/L, [RMgX]:[YRX] = 1:1 molar ratio. ^b After the completion of the addition of 1 at -30 °C, stirring was continued for the prescribed time interval at 0 °C. ^c After the completion of the addition of 1 at 0 °C, stirring was continued for the prescribed time interval at 20 °C. ^d [RMgX]:[YRX] = 1:0.5 molar ratio.

reaction with 3-bromopropyl chloride showed higher reactivity and selectivity with the bromide function. Mono-coupling was possible even in the reaction with 1,4-dibromobutane (no. 29). The selective couplings with halogen atom in the presence of other functional groups were possible, which, we think, provide attractive synthetic routes to 2-functionalized butadienes. Li₂CuCl₄ was found to be superior to CuI in this reaction also as a catalyst. Ester, cyano, ether, and hydroxy functions were not affected during the reaction. This coupling reaction can be applied to other simple Grignard reagents, whose results are also shown in Table I (no. 10-14). Even the vinyl Grignard reagent gave moderate yields. As shown above, the selective cross-coupling reaction of 1 with functionally substituted halides may find a wide application in the synthesis of 2-functionally substituted butadienes.

Experimental Section

Reagents. 1 was prepared as previously reported.¹³ CuI, Pd(PPh₃)₄, and 3-(*p*-iodophenyl)-1-iodopropane were prepared by the procedures described elsewhere.¹⁴⁻¹⁶ Commercial cupric chloride and lithium chloride were used after drying in vacuo. Li₂CuCl₄ was used as a mixture of [LiCl]:[CuCl₂] = 2:1 molar ratio.

Cross-Coupling Reaction of 1 with Alkyl Halides by Use of CuI. The reactions were carried out as described in the previous report.⁹

Cross-Coupling Reaction of 1 with Alkyl Halides, Aryl Halides, and Functional Halides by Use of Li₂CuCl₄. To a

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mixture of halide (0.1 mol), Li_2CuCl_4 (1-5 mol % to halide), and THF (50 mL) in 300-mL four-necked flask was added 1 (0.1 mol) in THF (100 mL) dropwise with stirring at the prescribed temperature under nitrogen atmosphere. An exothermic reaction occurred during the addition (in the cases of bromide, iodide), and the color of the contents gradually changed from reddish brown to black. After the completion of the addition, stirring was continued at the same temperature for the prescribed time interval.

The organic layer was separated after hydrolyzing the reaction mixture with 6 N HCl, and the aqueous layer was extracted with two portions of diethyl ether (100 mL). The combined organic extract was washed first with 5 % aqueous sodium hydrogen carbonate and then with water, dried (Na_2SO_4), and distilled. The reaction products were identified by comparing their IR, MS, and NMR spectra with the reported data.^{17,18} All the products gave reasonable elemental analyses.

Registry No. 1, 32657-89-9; $n\text{-C}_8\text{H}_{17}\text{Br}$, 111-83-1; $n\text{-C}_8\text{H}_{17}\text{I}$, 629-27-6; $n\text{-C}_8\text{H}_{17}\text{Cl}$, 111-85-3; $n\text{-C}_6\text{H}_{13}\text{CH}(\text{CH}_3)\text{Br}$, 557-35-7; $n\text{-C}_6\text{H}_{13}\text{CH}(\text{CH}_3)\text{I}$, 557-36-8; $\text{C}_6\text{H}_5\text{I}$, 591-50-4; $\text{C}_6\text{H}_5\text{Br}$, 108-86-1; $p\text{-BrC}_6\text{H}_4\text{I}$, 589-87-7; $p\text{-ClC}_6\text{H}_4\text{CH}_2\text{Br}$, 622-95-7; $p\text{-BrC}_6\text{H}_4\text{CH}_2\text{Br}$, 589-15-1; $p\text{-BrC}_6\text{H}_4\text{CH}_2\text{CH}_2\text{I}$, 85356-68-9; $p\text{-IC}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}_2\text{I}$, 85356-69-0; $\text{CH}_3\text{OCOCH}_2\text{CH}_2\text{I}$, 5029-66-3; $\text{C}_2\text{H}_5\text{OCO}(\text{CH}_2)_3\text{Br}$, 2969-81-5; $\text{CH}_3\text{OCO}(\text{CH}_2)_3\text{I}$, 14273-85-9; $\text{CH}_3\text{OCO}(\text{CH}_2)_5\text{I}$, 14273-91-7; $\text{ClCH}_2\text{CH}_2\text{CH}_2\text{Br}$, 109-70-6; $\text{BrCH}_2\text{CH}_2\text{CH}_2\text{Br}$, 110-52-1; $\text{C}_6\text{H}_5\text{COOCH}_2\text{CH}_2\text{I}$, 39252-69-2; $\text{HOCH}_2\text{CH}_2\text{I}$, 624-76-0; $\text{CH}_3\text{OCO}(\text{CH}_2)_4\text{I}$, 14273-88-2; $\text{NCCH}_2\text{CH}_2\text{CH}_2\text{Br}$, 5332-06-9; $\text{C}_6\text{H}_5\text{OCH}_2\text{CH}_2\text{Br}$, 589-10-6; CuI , 7681-65-4; Li_2CuCl_4 , 15489-27-7; $\text{Pd}(\text{PPh}_3)_4$, 14221-01-3; $i\text{-PrMgBr}$, 920-39-8; $\text{C}_6\text{H}_5\text{MgBr}$, 100-58-3; $\text{CH}_2=\text{CHMgBr}$, 1826-67-1; $\text{CH}_2=\text{CHC}(n\text{-C}_8\text{H}_{17})=\text{CH}_2$, 5732-02-5; $\text{CH}_2=\text{CHC}(\text{CH}(\text{CH}_3)\text{-}n\text{-C}_6\text{H}_{13})=\text{CH}_2$, 85356-70-3; $(\text{CH}_3)_2\text{CH}(\text{C}-\text{H}_2)_7\text{CH}_3$, 6975-98-0; $\text{C}_6\text{H}_5(\text{CH}_2)_7\text{CH}_3$, 2189-60-8; $\text{CH}_2=\text{CH}(\text{C}-\text{H}_2)_7\text{CH}_3$, 872-05-9; $\text{CH}_2=\text{CHC}(\text{C}_6\text{H}_5)=\text{CH}_2$, 2288-18-8; $\text{CH}_2=\text{CHC}(\text{C}_6\text{H}_4\text{Br-}p)=\text{CH}_2$, 38829-09-3; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{C}_6\text{H}_4\text{Cl-}p)=\text{CH}_2$, 85356-71-4; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{C}_6\text{H}_4\text{Br-}p)=\text{CH}_2$, 85356-72-5; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{C}_6\text{H}_4\text{I-}p)=\text{CH}_2$, 85356-73-6; $\text{CH}_2=\text{CHC}(\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}_2\text{I-}p)=\text{CH}_2$, 85356-74-7; $\text{CH}_2=\text{CHC}(\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}_2\text{I-}p)=\text{CH}_2$, 85356-82-7; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{COOC}_2\text{H}_5)=\text{CH}_2$, 38049-11-5; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{COOCH}_3)=\text{CH}_2$, 85356-75-8; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{COOCH}_3)=\text{CH}_2$, 85356-76-9; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{Cl})=\text{CH}_2$, 26831-14-1; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{CH}_2\text{Br})=\text{CH}_2$, 85356-77-0; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{OCOC}_6\text{H}_5)=\text{CH}_2$, 85356-78-1; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{OH})=\text{CH}_2$, 27974-99-8; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{COOCH}_3)=\text{CH}_2$, 85356-79-2; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{CN})=\text{CH}_2$, 85356-80-5; $\text{CH}_2=\text{CHC}(\text{CH}_2\text{CH}_2\text{OC}_6\text{H}_5)=\text{CH}_2$, 85356-81-6.

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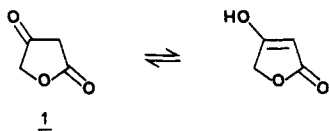
2,4(3H,5H)-Furandione: Heteroannulations with Aromatic *o*-Amino Carbonyl Compounds and Condensations with Some *vic*-Polyones¹

Diane Grob Schmidt, Paul D. Seemuth, and Hans Zimmer*

Department of Chemistry, University of Cincinnati,
Cincinnati, Ohio 45221

Received October 15, 1982

The chemical versatility of 2,4(3H,5H)-furandione (β -tetronic acid 1) and its synthetic applicability are of con-



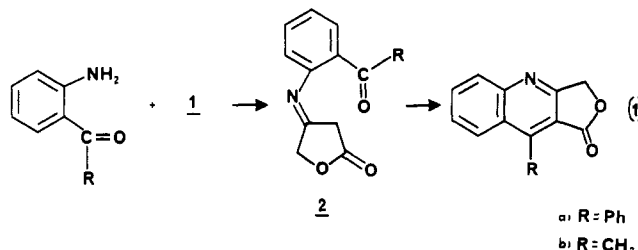
siderable current interest.² The multifunctional character

(1) (a) This is part 31 of the series "Substituted γ -Butyrolactones". (b) For part 30 of this series see: Amer, A.; Ventura, M.; Zimmer, H. *J. Heterocycl. Chem.* **1983**, *19*, 359.

of this small molecule confers an intriguing synthetic potential, which has prompted us to explore its chemistry. Recently, we reported the first facile synthesis of 3-(phenylmethylene)-2,4(3H,5H)-furandione by reaction of 1 with aromatic aldehydes^{2a} and their conversion into the novel ring system 2,5-dihydrofuro[3,4-d]-1,2-oxaphosphol-4-(6H)-one.^{2a} We now report the results of reactions of 1 with *o*-aminobenzophenones, aromatic *o*-amino aldehydes, and some 1,2,3-tricarbonyl compounds.

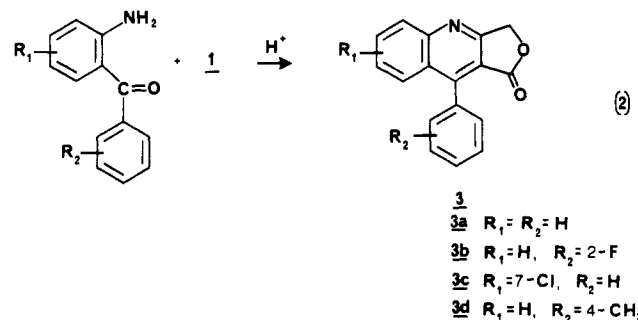
Results and Discussion

Heteroannulations of 1 with Aromatic *o*-Amino Carbonyl Compounds. Earlier workers³ reported that a condensation of 1 with 2-aminobenzophenone gave β -(2-benzoylphenylimino)- γ -butyrolactone (2, eq 1). Cy-



clization of 2 afforded type 3 compounds. The conversion that we report here seems, however, more convenient since it eliminates the preparation and isolation of the intermediate anil. Further, we have demonstrated that the condensation affords 3 with a variety of substituents.

The reaction of 1 with 1 equiv of a substituted 2-aminobenzophenone, in the absence of solvent and in the presence of concentrated HCl, effects in one pot a regioselective double condensation (eq 2) to form 3. This



condensation proceeds successfully with either electron-withdrawing or electron-releasing substituents on the phenyl rings. For the compounds studied, isolated yields

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