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Palladium-catalyzed reactions of vinyl bromides with disubstituted alkynes: a new synthesis of fulvenes

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Abstract

The palladium-catalyzed reaction of vinylic bromides with disubstituted acetylenes at 100 °C in the presence of triethylamine produces penta- or hexa-substituted fulvenes in low to moderate yields. Reactions with 3-hexyne under the same conditions usually yield dialkylidenecyclopentenes as products, apparently by rearrangement of the expected fulvenes. Fulvenes formed from the reactions of disubstituted alkynes with Z-2-bromovinyl ethyl ether can be hydrolyzed to produce cyclopentadiene carboxaldehydes.

Introduction

Fulvenes are a class of compounds which have proven useful both in natural product [1] and metallocene synthesis [2]. The preparation of these compounds has a long and varied history [3,4]. Fulvene derivatives are often prepared by the condensation of the cyclopentadienide derivative with carbonyls or other acceptors [3–5], or by Grignard reactions with cyclopentadienones followed by dehydration [3,4,6]. Cyclization methods are less common, and only a few examples of transition-metal catalyzed fulvene syntheses have been reported [7,8], the most notable being a nickel-catalyzed reaction of 1,1-diaryl-2,2-dibromoethenes with disububstituted alkynes [8] which produces hexaaryl-substituted fulvenes in low to moderate yield. The authors suggest that the alkynes react first with the nickel to produce a nickelacyclopentadiene intermediate, which then reacts with the dibromide. As part of our investigation into palladium reactions with disubstituted alkynes [9,10], we have discovered a new one-step method for preparing certain penta- and hexa-sub-

* Retired.

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stituted fulvenes [10] by a palladium-catalyzed reaction of vinyl bromides with disubstituted alkynes, and report here our investigation of this reaction.

Results and discussion

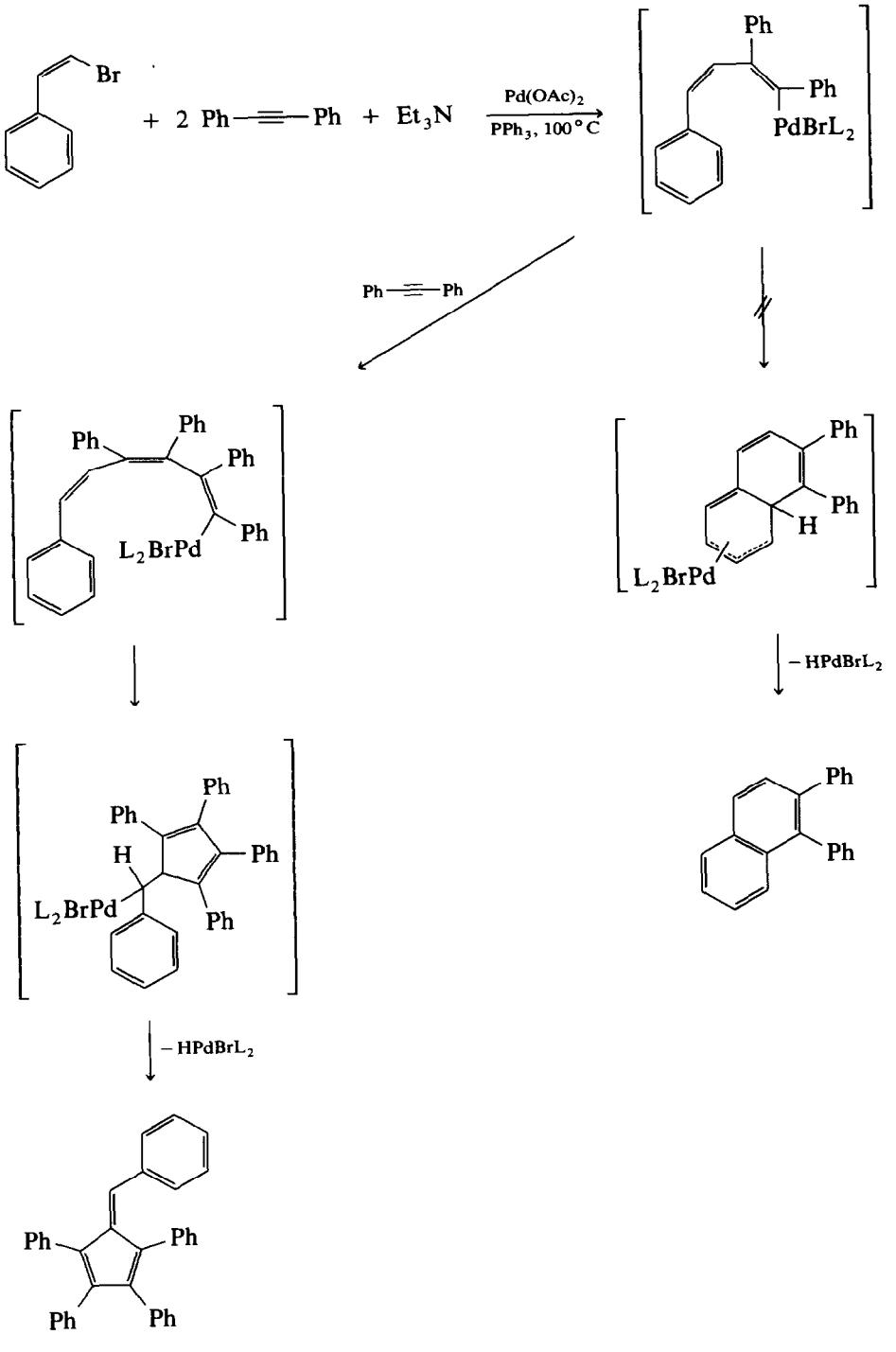
Previously, we had observed that the palladium-catalyzed reaction of aryl iodides with diphenylacetylene gives a naphthalene product derived from two moles of alkyne and one mole of aryl iodide. We carried out the palladium-catalyzed reaction of *Z*-1-bromo-2-phenylethene with diphenylacetylene ($Pd(OAc)_2$, PPh_3, Et_3N, 100 °C) with the expectation that one molecule of alkyne would insert and that the adduct would then cyclize to a naphthalene derivative by a similar mechanism [10]. Apparently, however, the addition of the second alkyne molecule to the 1 : 1 complex is much faster than the naphthalene ring closure. Instead of the naphthalene, we obtained 1,2,3,4,6-pentaphenylfulvene. The structure of the product was established by X-ray crystallography [10]. We found no trace of naphthalene products in our reaction mixtures. The proposed mechanism is shown in Scheme 1.

Other successful fulvene syntheses by this method are summarized in Table 1. The stereochemistry of the vinyl bromide is not important for the fulvene formation. *E*-1-bromo-2-(*p*-methoxyphenyl)ethene, 1-bromo-1-propene, *E*-methyl 3-bromo-2-methylpropenoate, and *Z*-2-bromovinyl ethyl ether all reacted to give the fulvenes. However, vinyl bromide, 2,2-diphenylbromoethene, and *Z*-1-bromo-2-(*m*-nitrophenyl)ethene all failed to give anything but intractable oils when reacted with diphenylacetylene.

Diarylacetylenes consistently gave the fulvenes. Diphenylacetylene and di-*p*-anisylacetylene reacted normally. The unsymmetrical alkyne phenyl(*p*-anisyl)acetylene also reacted with *E*-1-bromo-2-(*p*-methoxyphenyl)ethene as expected, but gave a mixture of all possible isomeric fulvenes judging from the NMR spectrum of the reaction product. Di-(*p*-nitrophenyl)acetylene gave only oligomers in reactions with *E*-1-bromo-2-(*p*-methoxyphenyl)ethene, and dimethyl acetylenedicarboxylate polymerized very rapidly in the reaction mixture.

Reactions of 3-hexyne with 1-bromo-1-propene, and *Z*-1-bromo-2-phenylethene produced viscous red oils. The products were very difficult to separate, but GC-MS showed that the products had many peaks of the molecular weights expected for the fulvenes. ^1H NMR spectra, however, indicated that fulvenes were not significant products.

We felt that the desired fulvenes might be forming initially, but were readily isomerized. This was confirmed by the reaction of *E*-1-bromo-2-(*p*-methoxyphenyl)-ethene with 3-hexyne, which gave initially an orange-red oil whose ^1H NMR showed at least five different ethyl groups and two methoxy singlets. The NMR sample in chloroform-*d* changed in two hours to a yellow product with only three types of ethyl groups and one methoxyl. The same reaction did not occur in methanol-d_4. The reaction was scaled up and the product mixture was isomerized in methylene chloride at room temperature. Careful recrystallization from methanol gave the product as a white solid (m.p. 36.0–37.5 °C). The ^1H and ^{13}C NMR spectra indicate that the product is a *p*-methoxybenzilidene-ethylidene-triethylcyclopentene. The 1,3-isomer is most probable because of the more favorable conjugation of the double bonds (Eq. 1). The initial orange-red product following workup may be a



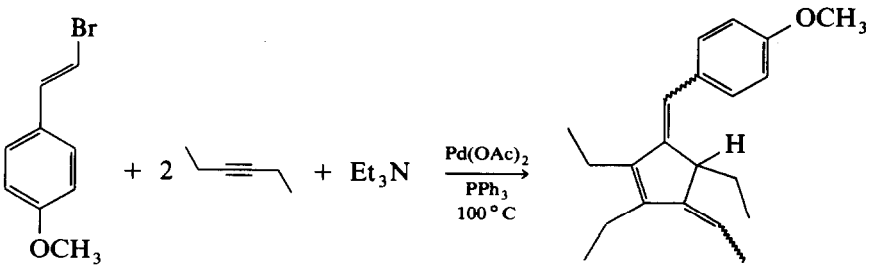
Scheme 1. Proposed mechanism of fulvene formation.

Table 1

Fulvene syntheses

Vinyl bromide	Alkyne	Reaction solvent and time, 100 °C [a]	Product	Yield (%)
Z-PhCH=CHBr [b]	PhC≡CPh	CH_3NO_2, 24 h	1,2,3,4,5-Pentaphenylfulvene	27
E-*p*-AnCH=CHBr	PhC≡CPh	CH_3NO_2, 15 h [c]	5-*p*-Anisyl-1,2,3,4-tetraphenylfulvene	43
E-*p*-AnCH=CHBr	*p*-AnC≡CAn-*p*	CH_3NO_2, 6 h [c]	1,2,3,4,5-Penta-*p*-anisylfulvene	59
E-$CH_3O_2CC(CH_3)$=CHBr	PhC≡CPh	$(C_2H_5)_3N$, 15 h [c]	5-Methoxycarbonyl-5-methyl-1,2,3,4-tetraphenylfulvene	12
E-Ch_3CH=CHBr	*p*-AnC≡CAn-*p*	$(C_2H_5)_3N$, 19 h [c]	1,2,3,4-Tetra-*p*-anisyl-5-methylfulvene	30
Z-C_2H_5OCH=CHBr	PhC≡CPh	$(C_2H_5)_3N$, 24 h [c]	5-Ethoxy-1,2,3,4-tetraphenylfulvene	31
Z-C_2H_5OCH=CHBr	$C_2H_5C{\equiv}CC_2H_5$	$(C_2H_5)_3N$, 29 h [c]	5-Ethoxy-1,2,3,4-tetraethylfulvene	26 [d]

[a] Reaction conditions: 2.5 mmol of the vinyl bromide, 5.5 mmol alkyne, 0.05 mmol $Pd(OAc)_2$, 0.10 mmol PPh_3 in 2 ml $(C_2H_5)_3N$ and 3 ml of solvent; heated at 100 °C in capped tubes with stirring. Concentration of the vinyl bromide and alkyne were twice the above in the ethoxyvinyl bromide reactions. [b] About 85% *Z* and 15% *E* isomer. [c] Tri-*o*-tolylphosphine used instead of triphenylphosphine. [d] Yield of aldehyde obtained after hydrolysis.

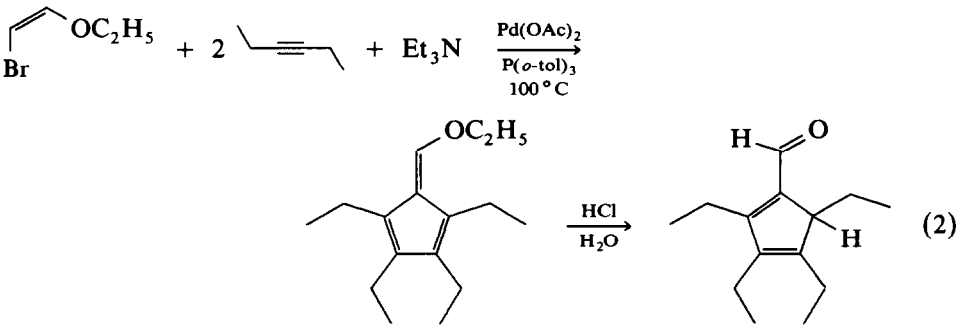


(1)

mixture of stereoisomers. The products were isomerized during gas chromatography also, and only one peak was observed. The yield by GLC with an internal standard (triphenylmethane) was 83%.

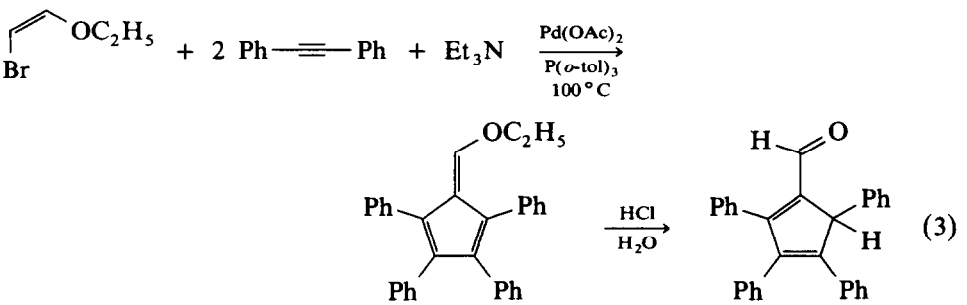
It is not clear how the trienes are formed, but palladium hydride eliminations and readditions around the ring and on the side chains could explain the reaction. This would not occur in the case of the diarylacetylenes since the phenyl carbon which is bonded to the fulvene ring would not have a β-hydride which could eliminate.

Although most reactions with 3-hexyne appeared to give the trienes, *Z*-2-bromovinyl ethyl ether and 3-hexyne reacted to give the expected fulvene. The crude liquid product was hydrolyzed with aqueous hydrochloric acid to the cyclopentadiene carboxaldehyde in 26% overall yield (Eq. 2).



(2)

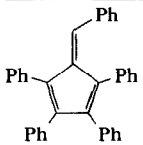
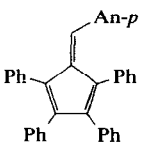
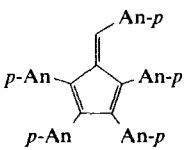
Z-2-bromovinyl ethyl ether reacted with diphenylacetylene in similar fashion to give the crystalline fulvene in 31% yield. Hydrolysis with aqueous hydrochloric acid gave the orange, crystalline dienal in 55% yield (Eq. 3). Thus a simple route is provided to tetrasubstituted cyclopentadiene carboxaldehydes.

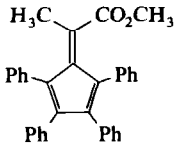
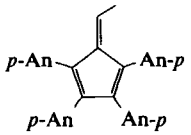
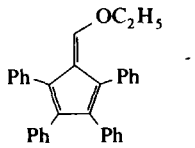


(3)

Table 2

Properties

Fulvene	M.p.	Mol. wt (HRMS) Mol. wt (HRMS)		^{1}H NMR ($CDCl_3$, ppm)	^{13}C NMR ($CDCl_3$, ppm)
		Found	Calcd.		
	201–202 °C (200–201 °C) [a,c]			7.42 (s, 1H), 7.28–7.23 (m, 3H) 7.04–6.77 (m, 22H)	145.9, 144.3, 141.7, 141.3, 141.2, 136.6, 136.1, 135.6, 135.3, 135.2, 131.6, 131.1, 130.8, 130.5, 130.4, 130.3, 127.7, 127.6, 127.3, 127.2, 127.1, 127.0, 126.5, 126.3, 126.2, 125.4
	207–208 °C (199–199 °C) [b] (197–198 °C) [c]	488.22	488.214	7.36 (s, 1H), 7.35–6.75 (m, 22H), 6.40 (d, $J = 9$, 2H), 3.65 (s, 3H)	159.6, 145.6, 142.8, 141.4, 141.1, 137.1, 136.4, 135.6, 135.5, 132.6, 131.7, 130.9, 130.6, 130.3, 128.2, 127.7, 127.4, 127.2, 127.1, 126.4, 126.2, 126.1, 125.4, 112.7, 55.1
	190–191 °C	608.256	608.256	7.28 (s, 1H), 7.14 (d, $J = 8$, 2H), 6.90–6.30 (m, 20H), 3.79 (s, 3H), 3.69 (s, 3H), 3.68 (s, 3H), 3.67 (s, 6H)	159.3, 158.1, 157.9, 157.8, 157.5, 139.9, 135.1, 132.7, 132.3, 132.0, 131.8, 131.6, 129.8, 128.5, 128.2, 128.1, 113.3, 113.1, 112.8, 112.6, 55.1, 54.9

	181–182 °C	454.193	454.193	7.20 (m, 10H), 6.95 (m, 6H), 6.80–6.72 (m, 4H), 2.92 (s, 3H), 1.82 (s, 3H)	170.2, 144.9, 140.8, 138.1, 136.0, 135.0, 134.9, 133.6, 131.2, 130.5, 130.0, 129.9, 128.1, 127.3, 127.1, 126.7, 126.4, 51.4, 20.3
	149.5–151.5 °C	517.237	517.238	7.17 (d, $J = 9$, 2H), 7.09 (d, $J = 9$, 2H), 6.80 (m, 8H), 6.58 (dd, $J = 2$ and 10, 4H), 6.44 (q, $J = 8$, 1H), 3.78 (s, 6H), 3.68 (s, 3H), 3.67 (s, 3H), 1.68 (d, $J = 8$, 3H)	158.2, 158.1, 157.8, 157.7, 145.9, 144.3, 139.5, 139.2, 133.8, 132.4, 131.8, 131.5, 131.4, 130.7, 128.3, 128.2, 128.1, 113.3, 113.2, 112.8, 112.6, 55.07, 54.9, 16.2
	204–205 °C	426.198	426.198	7.27–7.12 (m, 10H), 7.03–6.98 (m, 7H), 6.87–6.81 (m, 4H), 3.94 (q, $J = 7.1$, 2H), 1.14 (t, $J = 7.1$, 3H)	157.9, 141.3, 138.3, 137.4, 136.2, 136.1, 135.8, 134.4, 131.4, 131.1, 130.7, 130.6, 129.5, 127.9, 127.2, 127.0, 126.6, 126.3, 125.8, 125.7, 124.1, 71.4, 15.0

[a] Reported m.p. by A. Sekiguchi, H. Tanikawa and W. Ando, *Organometallics*, 4 (1985) 584. [b] D. Taber, N. Picus, E.I. Becker and P.E. Spoerri, *J. Am. Chem. Soc.*, 77 (1955) 1010. [c] D. Dilthey and P. Hucteman, *J. Prakt. Chem.*, 154 (1940) 238.

Experimental

Z-1-bromo-2-phenylethene, *Z*-1-bromo-2-(*m*-nitrophenyl)ethene (88% yield, m.p. 188–189 °C) and *E*-1-bromo-2-(*p*-methoxyphenyl)ethene were prepared from the corresponding cinnamic acid dibromides and aqueous sodium bicarbonate [11,12]. *E*-methyl 3-bromo-2-methylpropenoate [13] was prepared by the literature method. The 1-bromo-1-propene (Aldrich) was a mixture of *E* and *Z* isomers. GLC analysis was done using a 10 foot SE-30 column.

General procedure for the reaction of vinyl bromides with disubstituted alkynes

A mixture of 2.5 mmol of the vinyl bromide, 5.5 mmol of the alkyne, 2 ml of triethylamine, 3 ml of solvent (or additional amine), 0.05 mmol of palladium acetate and 0.10 mmol of triphenyl- or tri(*o*-tolyl)phosphine was prepared in a heavy-walled pyrex tube and sealed with a rubber septum and holed metal bottle cap. The mixture was warmed with shaking until homogeneous and then heated at 100 °C in a steam bath for 6–24 h as indicated in Table 1. The reaction mixture was diluted with methylene chloride, filtered through Celite to remove palladium metal and then the solution was washed several times with cold 6 *N* hydrochloric acid and finally with aqueous sodium bicarbonate. The products were separated by chromatography on silica and generally recrystallized from hexane–methylene chloride. The properties and spectra of the products prepared appear in Table 2.

*Di-(*p*-anisyl)acetylene*

To 34 ml of 1.6 *M* n-butyllithium in hexane at −25 °C was added 5.5 g of isopropylamine. 22 ml of THF was added and the mixture stirred at −25 °C. 15 g (54.6 mmol) of 1,2-di-(*p*-anisyl)-1-chloroethene [14] was added with stirring. The solution was allowed to warm up to 15 °C and it was stirred at this temperature for 1.5 h. Then 20 ml of cold 3 *N* hydrochloric acid was added. A precipitate of the product formed. This was separated by filtration, washed with water and recrystallized from methanol. There was obtained 9.75 g (75%) of off-white crystals of the alkyne, m.p. 151–152 °C (reported m.p. 142 °C [14]). ^{1}H NMR ($CDCl_3$, ppm): 7.44 (d, $J = 8.9$, 4H); 6.85 (d, $J = 8.9$, 4H); 3.79 (s, 6H).

Reaction of E*-1-bromo-2-(*p*-methoxyphenyl)ethene with 3-hexyne*

A large pressure bottle was charged with a stir bar, 0.099 g (0.4 mmol) of palladium acetate, 0.243 g (0.8 mmol) of tri-(*o*-tolyl)phosphine, 2.5 g (0.0124 mol) of *E*-1-bromo-2-(*p*-methoxyphenyl)ethene, 2.13 g (0.026 mol) of 3-hexyne, and 15 ml of triethylamine and capped. The mixture was warmed and shaken until homogeneous, then heated at 100 °C for 18 h. The reaction mixture was poured into a separatory funnel with water and extracted with methylene chloride. The mixture was dried over magnesium sulfate, then chromatographed on silica with pentane to give a red oil. 6 ml of methylene chloride was added and the solution was allowed to sit at room temperature overnight. The now dark orange solution was concentrated and then recrystallized in methanol by seeding and leaving in the freezer overnight. White, fluffy solid was obtained in 7.5% yield, m.p. 36–37.5 °C. Yield by GLC with triphenylmethane as internal standard was 83%. HRMS: 296.215 (calc.: 296.214). ^{1}H NMR ($CDCl_3$, ppm): 7.40 (d, $J = 8.8$, 2H); 6.83 (d, $J = 8.8$, 2H); 6.18 (s, 1H); 5.40 (q, $J = 5.8$, 1H); 4.09 (s, 1H); 3.75 (s, 3H); 2.30 (m, 4H); 1.84 (d, $J = 7.2$, 3H);

1.70 (m, 2H); 1.09 (t, $J = 7.5$, 3H); 1.05 (t, $J = 7.5$, 3H); 0.54 (t, $J = 7.3$, 3H). ^{13}C NMR: 157.6, 147.0, 146.8, 146.0, 145.7, 131.3, 129.4, 114.7, 113.9, 110.5, 55.2, 41.8, 23.2, 18.3, 18.1, 14.6, 13.79, 13.76, 7.9.

2,3,4,5-Tetraphenyl-1,3-cyclopentadiene carboxaldehyde

A 100 ml roundbottom flask was charged with a stir bar, 0.600 g (1.44 mmol) of 5-ethoxy-1,2,3,4-tetraphenylfulvene and 30 ml of ether. The nonhomogeneous mixture was topped with a reflux condenser and then 0.25 ml (2 equivalents) of conc. HCl was added. The mixture was stirred vigorously for 45 min. Most of the solid had dissolved. The solution was poured into a separatory funnel with ether and water and the aqueous layer drained off. The organic layer was washed with water, then saturated aqueous $NaHCO_3$, then water twice. The ether layer was dried with magnesium sulfate and evaporated to a red oil. Chromatography on silica with 7.5% ethyl acetate/petroleum ether gave an orange solid. Recrystallization in ethyl acetate/petroleum ether gave 0.294 g dienal, m.p. 158.5–160 °C. HRMS: 398.16706 (calc.: 398.16632). ^1H NMR ($CDCl_3$, ppm): 9.63 (s, 1H); 7.3–7.0 (m, 20H); 5.23 (s, 1H). ^{13}C NMR: 186.9, 162.9, 154.8, 145.2, 143.2, 136.5, 134.4, 134.2, 132.4, 130.0, 129.8, 129.3, 128.7, 128.5, 128.3, 128.1, 128.0, 127.8, 127.5, 126.8, 57.9.

2,3,4,5-Tetraethyl-1,3-cyclopentadiene carboxaldehyde

A mixture of 0.135 g (0.6 mmol) of palladium acetate, 0.366 g (1.2 mmol) of tri(*o*-tolyl)phosphine, 2.265 g (15 mmol) of *Z*-2-bromovinyl ethyl ether, 2.722 g (33 mmol) of 3-hexyne and 15 ml of triethylamine was sealed in a pressure vial, stirred 5 min at room temperature, and then heated at 100 °C for 17 h. The mixture was dissolved in ethyl acetate, washed three times with water, and back extracted once with ethyl acetate. The combined organics were dried over magnesium sulfate and concentrated to a red oil.

The crude fulvene was transferred to a 100 ml roundbottom flask with a stir bar. 35 ml of ether was added, and then 1.25 ml (15 mmol) of conc. HCl. Black material immediately precipitated. The mixture was stirred vigorously for 25 min. The mixture was poured into a separatory funnel with ether and water. The ether layer was washed twice with water, once with saturated aqueous $NaHCO_3$, and twice more with water. The organic layer was vacuum filtered to remove a yellow solid, and the clear orange filtrate was dried over magnesium sulfate and chromatographed on silica with 4% ethyl acetate/petroleum ether to give 0.788 g (26%) orange oil. ^1H NMR ($CDCl_3$, ppm): 9.98 (s, 1H); 3.44 (t, $J = 4.5$, 1H); 2.73 (q, $J = 7.6$, 2H); 2.50 (sextet, $J = 7.4$, 1H); 2.36–2.04 (m, 5H); 1.21 (t, $J = 7.6$, 3H); 1.09 (t, $J = 7.6$, 3H); 1.05 (t, $J = 7.6$, 3H); 0.37 (t, $J = 7.4$, 3H). ^{13}C NMR: 184.0, 169.3, 158.9, 142.6, 139.5, 50.2, 20.9, 19.9, 18.9, 17.8, 16.1, 15.1, 14.5, 7.05.

Note added in proof. While this paper was in press, a related study appeared (G.C.M. Lee, B. Tobias, J.M. Holmes, D.A. Harcourt and M.E. Garst, J. Am. Chem. Soc., 112 (1990) 9330). The authors apparently were not aware of our prior work (refs. 9f, 10).

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