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Polybromocyclopentadienes in Diels–Alder Reactions. Reactions of Hexabromo- and 5,5-Dimethoxytetrabromo-1,3-cyclopentadienes with *para*-Substituted Allyl Benzoates

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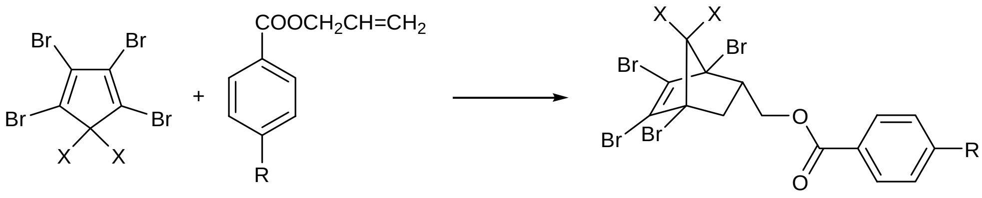
Abstract—The Diels–Alder reaction of hexabromo- and 5,5-dimethoxytetrabromo-1,3-cyclopentadienes with *para*-substituted allyl benzoates occurs with high stereoselectivity, yielding the corresponding *endo* adducts. The kinetics of this reaction were studied, and its activation parameters were determined. The reaction is favored by the presence of both electron-donor and electron-acceptor substituents in the aromatic ring. The diene–dienophile system was assumed to react according to the “neutral” pattern.

Increased interest in Diels–Alder reactions involving polybromocyclopentadienes is explained by both wide synthetic potential of these reactions which allow preparation of difficultly accessible bicyclic polybrominated polyfunctional compounds and high reliability of quantitative parameters of the process which is not usually accompanied by side reactions.

Polybromocyclopentadienes are fairly reactive in conventional Diels–Alder reactions. This follows from the high yields in reations with typical electron-acceptor dienophiles, such as maleic anhydride, *N*-arylmaleimides, *N*,*N*-bismaleimides, *p*-benzoquinone, etc. [1–4]. Presumably, their reactivity originates from the dual effect of bromine atoms attached to the carbon skeleton. On the one hand, negative inductive effect of bromine atoms reduces the energy of the lowest unoccupied molecular orbital (LUMO), thus favoring reversed Diels–Alder reaction. On the other hand, conjugation between lone electron pairs on the bromine atoms and π-bonds should increase the energy of the highest occupied molecular orbital (HOMO) of the diene, which is responsible for its ability to react with electron-acceptor dienophiles. However, kinetic studies showed relatively low reactivity of hexabromo-1,3-cyclopentadiene and 5,5-dimethoxytetrabromo-1,3-cyclopentadiene in Diels–Alder reactions, as compared to unsubstituted cyclopentadiene [2, 4–8]. The general reaction pattern does not change on replacement of hydrogen atoms in the cyclopentadiene molecule by bromine. Therefore, we assumed that the reactivity of hexabromo-1,3-cyclopentadiene and 5,5-dimethoxytetrabromo-1,3-cyclopentadiene can be treated in terms of the generally accepted principles of the mechanism of Diels–Alder reaction [2–8].

We previously [2, 4–7] studied reactions of hexabromo- and 5,5-dimethoxytetrabromo-1,3-cyclopentadienes with various dienophiles, examined the stereochemistry and kinetic relations holding in these reactions, and revealed factors determining the reactivity of the diene–dienophile systems. We found that within a single reaction series, replacement of the two geminal bromine atoms in hexabromo-1,3-cyclopentadiene by methoxy groups leads to change of the reaction pattern from the reversed (where diene acts as acceptor, and dienophile, as donor) to normal (where diene is donor, and dienophile is acceptor) [4–7]. We were the first to obtain experimental proofs for the occurrence of a “neutral” type of donor–acceptor interaction in reactions of hexabromo- and 5,5-dimethoxytetrabromo-1,3-cyclopentadienes with arylacrylates and arylacrylamides [6, 7]. It was shown that

Scheme 1.



I, **II** **IIIa**–**IIIf** **IVa**–**IVf**, **Va**–**Vf**

I, **IV**, X = Br; **II**, **V**, X = CH_3O; **III**–**V**, R = *p*-CH_3O (**a**), *p*-CH_3 (***b***), H (**c**), *p*-Cl (**d**), *p*-Br (**e**), *p*-O_2N (**f**).

the reactivity of hexabromo-1,3-cyclopentadiene with respect to various dienophiles is determined by parallel variations of the energy of donor–acceptor interaction and energy of localization [4] and that the reactivity of 5,5-dimethoxytetrabromo-1,3-cyclopentadiene in analogous reaction series depends on the donor–acceptor interaction of the addends; this was confirmed by the results of kinetic and thermochemical studies of the model Diels–Alder reactions between the above dienes and aryldihydrotriazolediones [2, 4–7].

With the goal of further studying factors responsible for the reactivity of addends in reactions with participation of hexabromo-1,3-cyclopentadiene (**I**) and 5,5-dimethoxytetrabromo-1,3-cyclopentadiene (**II**), in the present work we examined Diels–Alder reactions of compounds **I** and **II** with *para*-substituted allyl benzoates **IIIa**–**IIIf** (Scheme 1).

Intermolecular donor–acceptor interaction is one of the electronic factors determining the reactivity of addends in Diels–Alder reactions. The energy of donor–acceptor interaction depends on donor–acceptor parameters of the addends: In reactions involving diene as donor (or acceptor) and dienophile as acceptor (or donor), electron-donor (electron-acceptor) dienes and electron-acceptor (electron-donor) dienophiles are characterized by high reactivity [2, 9–14]. To confirm the above stated, we estimated the electron-acceptor power of dienophiles **IIIa**–**IIIf** by polarography. It is known that a measure of the electron-acceptor power is electron affinity [10]. The electron affinities of dienophiles **IIIa**–**IIIf** were determined according to the following equation [15]:

$$E_A = 1.04E_{1/2} + 1.39.$$

Here, E_A is the electron affinity of a dienophile, and $E_{1/2}$ is the half-wave reduction potential relative to a saturated calomel electrode. The results are given in Table 1.

The kinetics of the Diels–Alder reactions of compounds **I** and **II** with dienophiles **IIIa**–**IIIf** were studied by polarography, following decrease of the diene concentration in the reaction mixture. We preliminarily examined the polarographic behavior of dienes **I** and **II** and allyl benzoates **IIIa**–**IIIf** at a dropping mercury electrode in DMF using tetraethylammonium iodide as supporting electrolyte. It was found that the presence of dienophiles **IIIa**–**IIIf** and final products **IVa**–**IVf** and **Va**–**Vf** in the mixture does not interfere with polarographic determination of the current concentrations of dienes **I** and **II**.

The rate constants were calculated as described in [16], using kinetic equations for bimolecular reactions [17]. The relations between the logarithms of the rate constants and reciprocal temperature were linear, indicating that the reactions under study fit the Arrhenius equation, from which we determined the kinetic and thermodynamic activation parameters. The rate constants for the Diels–Alder reactions of dienes **I** and **II** with dienophiles **IIIa**–**IIIf** and the

Table 1. Half-wave reduction potentials ($E_{1/2}$) and electron affinities (E_A) of *para*-substituted allyl benzoates **IIIa**–**IIIf**

Compound	$E_{1/2}$	E_A, eV
Allyl *p*-methoxybenzoate (**IIIa**)	–1.57	–0.24
Allyl *p*-methylbenzoate (**IIIb**)	–1.49	–0.16
Allyl benzoate (**IIIc**)	–1.41	–0.07
Allyl *p*-chlorobenzoate (**IIId**)	–1.29	0.05
Allyl *p*-bromobenzoate (**IIIe**)	–1.27	0.05
Allyl *p*-nitrobenzoate (**IIIf**)	–0.93	0.42
Hexabromo-1,3-cyclopentadiene (**I**)[a]	–	0.73
5,5-Dimethoxytetrabromo-1,3-cyclopentadiene (**II**)[a]	–	≤–0.303

[a] Data of [2].

Table 2. Kinetic parameters of the Diels–Alder reactions of hexabromo-1,3-cyclopentadiene (**I**) and 5,5-dimethoxytetrabromo-1,3-cyclopentadiene (**II**) with *para*-substituted allyl benzoates **IIIa**–**IIIf** in chlorobenzene

Reactants	$k \times 10^6$, l mol^{-1} s^{-1}			E_a, kJ/mol	log$A$	$\Delta H^{\neq}$, kJ/mol	$-\Delta S^{\neq}$, J mol^{-1} K^{-1}
	100°C	110°C	120°C				
I, **IIIa**	46.4	81.7	138.0	69	5.3	65.8	194.5
I, **IIIb**	42.8	74.6	126.6	70.8	5.5	67.4	192.8
I, **IIIc**	37.4	59.4	97.8	74.5	5.9	71.3	189.0
I, **IIId**	42.0	73.7	122.0	70.8	5.5	67.4	192.8
I, **IIIe**	41.7	72.9	118.5	74.5	6.0	71.3	189.0
I, **IIIf**	57.7	93.5	163.5	63.2	4.6	60.0	200.4
II, **IIIa**	106.1	152.5	237.9	57.3	4.0	54.1	206.2
II, **IIIb**	89.7	143.1	215.9	59.4	4.2	56.2	204.2
II, **IIIc**	67.5	110.0	172.7	67.0	5.2	63.8	196.6
II, **IIId**	92.4	140.5	226.2	63.2	4.8	60.0	200.4
II, **IIIe**	91.0	138.8	218.1	60.2	4.4	57.1	203.3
II, **IIIf**	111.3	171.7	270.5	53.6	3.5	50.4	209.9

corresponding activation parameters are collected in Table 2. It is seen that these reactions are characterized by low energies of activation and high negative entropies of activation. These data suggest that the cycloaddition follows a concerted mechanism.

Diene **II** possessing a lower electron affinity than **I** is more reactive as diene toward dienophiles **IIIa**–**IIIf** (Table 2). This may be due to greater energy of donor–acceptor interaction, as well as to lower energy of π-electron delocalization. According to the data in Table 2, the reactivity of dienes **I** and **II** with respect to dienophiles **IIIa**–**IIIf** increases on introduction of both electron-donor and electron-acceptor substituents into the aromatic ring of the dienophile. These trends are illustrated by Figs. 1 and 2 where the relative reactivity parameters log(k_R/k_H) are plotted against the Hammett constants σ.

Our results led us to conclude that the process under study can be regarded as a neutral type of the Diels–Alder reaction, where the energy of stabilization

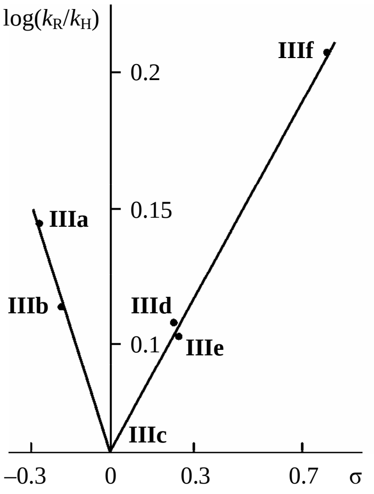


Fig. 1. Correlation between the relative reactivities [log(k_R/k_H)] of *para*-substituted allyl benzoates **IIIa**–**IIIf** in the Diels–Alder reaction with hexabromo-1,3-cyclopentadiene (**I**) (chlorobenzene, 110°C) and Hammett constants σ.

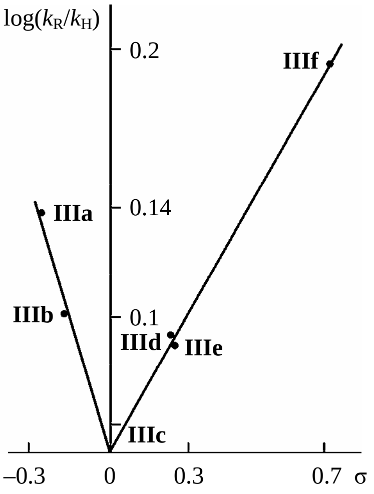
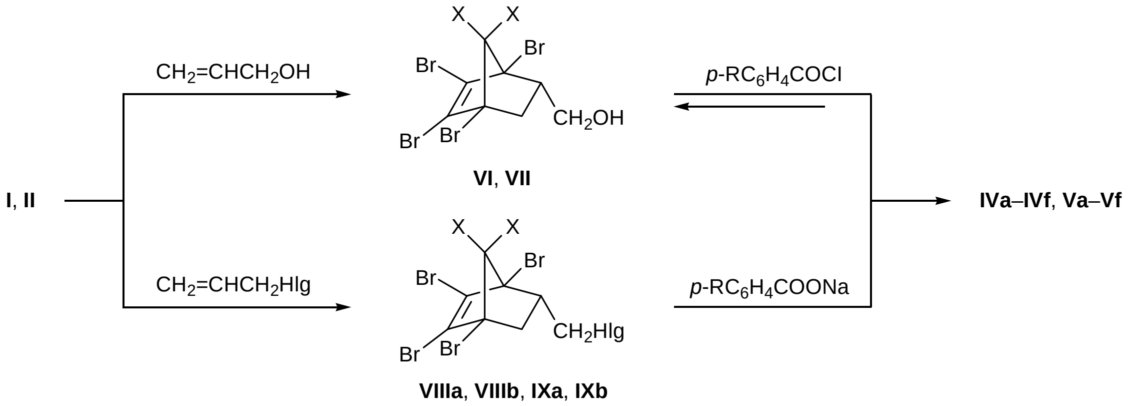


Fig. 2. Correlation between the relative reactivities [log(k_R/k_H)] of *para*-substituted allyl benzoates **IIIa**–**IIIf** in the Diels–Alder reaction with 5,5-dimethoxytetrabromo-1,3-cyclopentadiene (**II**) (chlorobenzene, 110°C) and Hammett constants σ.

Scheme 2.



I, **IV**, **VI**, **VIII**, X = Br; **II**, **V**, **VII**, **IX**, X = CH_3O; **III**–**V**, R = *p*-CH_3O (**a**), *p*-CH_3 (***b***), H (**c**), *p*-Cl (**d**), *p*-Br (**e**), *p*-O_2N (**f**);
VIII, **IX**, Hlg = Cl (**a**), Br (**b**).

Table 3. Yields, melting points, R_f values, and elemental analyses of compounds **IVa**–**IVf**, **Va**–**Vf**, and **VII**–**IX**

Comp. no.	Yield, %	mp, °C	R_f	Found, %			Formula	Calculated, %		
				C	H	Br		C	H	Br
IVa	72	103–104	0.90	25.41	1.22	67.96	$C_{15}H_{10}Br_6O_2$	25.66	1.42	68.35
IVb	73	97–98	0.90	26.67	1.30	66.72	$C_{16}H_{12}Br_6O_2$	26.83	1.68	67.01
IVc	78	91–92	0.91	25.95	1.38	65.23	$C_{16}H_{12}Br_6O_3$	26.25	1.64	65.53
IVd[a]	73	107–108	0.87	25.42	0.95	63.87	$C_{16}H_9Br_6ClO_2$	25.67	1.2	64.11
IVe	71	111–112	0.88	24.07	0.90	70.24	$C_{16}H_9Br_7O_2$	24.23	1.13	70.59
IVf[b]	77	180 (decomp.)	0.91	25.11	1.59	62.87	$C_{16}H_9Br_6NO_4$	25.31	1.86	63.21
IV	63	72–73	0.92	15.89	0.74	79.88	$C_8H_6Br_6O$	16.06	1.0	80.24
VIII[c]	59	107–108	0.88	15.33	0.60	77.49	$C_8H_5Br_6Cl$	15.58	0.81	77.84
IXa	56	119–120	0.88	14.37	0.55	84.40	$C_8H_5Br_7$	14.53	0.75	84.70
Va	81	141–142	0.93	35.54	2.37	52.68	$C_{17}H_{16}Br_4O_4$	33.79	2.65	52.95
Vb	84	150–151	0.92	34.75	2.65	51.34	$C_{18}H_{18}Br_4O_4$	34.97	2.91	51.75
Vc	83	123–124	0.93	33.87	2.59	50.07	$C_{18}H_{18}Br_4O_5$	34.09	2.84	50.44
Vd[d]	81	135–136	0.91	31.78	2.0	49.70	$C_{17}H_{15}Br_4ClO_4$	31.97	2.35	50.09
Ve	79	142–143	0.91	29.67	1.95	58.29	$C_{17}H_{15}Br_5O_4$	29.82	2.19	58.54
Vf[e]	84	163 (decomp.)	0.92	31.29	2.09	48.91	$C_{17}H_{15}Br_4NO_6$	31.45	2.31	49.28
VII	67	90–91	0.94	23.84	2.13	63.68	$C_{10}H_{12}Br_4O_3$	24.02	2.4	63.97
VIIIb[f]	61	124–125	0.90	22.95	1.89	61.39	$C_{10}H_{11}Br_4ClO_2$	23.16	2.12	61.69
IXb	59	130–131	0.90	21.09	1.56	70.81	$C_{10}H_{11}Br_5O_2$	21.33	1.95	71.02

[a] Found, %: Cl 4.63. Calculated, %: Cl 4.74.
[b] Found, %: N 1.71. Calculated, %: N 1.84.
[c] Found, %: Cl 5.76. Calculated, %: Cl 5.49.
[d] Found, %: Cl 5.21. Calculated, %: Cl 5.35.
[e] Found, %: N 1.84. Calculated, %: N 2.16.
[f] Found, %: Cl 6.61. Calculated, %: Cl 6.84.

Table 4. ^{1}H NMR and IR spectra of compounds **IVa**–**IVf** and **Va**–**Vf**

Comp. no.	^{1}H NMR spectrum, δ, ppm						*J*, Hz			IR spectrum, ν, cm^{-1}
	H_A	H_B	H_C	CH_2	OCH_3	H_{arom}	J_{AB}	J_{AC}	J_{BC}	
IVa	2.60	2.80	3.90	4.20	–	6.85–7.35 m (5H)	12.0	5.0	8.0	1775 (C=O), 1575 C=C)
IVb[a]	2.60	2.80	3.85	4.20	–	6.80–7.20 m (4H)	12.0	5.0	8.0	1765 (C=O), 1585 (C=C)
IVc[a]	2.60	2.75	3.75	4.25	–	6.80–7.35 m (4H)	12.0	5.0	8.0	1765 (C=O), 1575 (C=C), 1180 (C–O–C)
IVd	2.65	2.80	3.90	4.20	–	6.70–7.40 m (4H)	12.5	4.5	9.0	1770 (C=O), 1575 (C=C), 750 (C–Cl)
IVe	2.65	2.80	3.90	4.20	–	6.80–7.40 m (4H)	12.0	5.0	8.0	1760 (C=O), 1575 (C=C), 560 (C–Br)
IVf	2.60	2.80	3.90	4.20	–	6.80–7.50 m (4H,)	12.5	4.5	9.0	1780 (C=O), 1575 (C=C), 1355 (NO_2)
Va	2.30	2.50	3.40–3.55	4.30	3.60 s, 3.67 s	6.85–7.30 m (4H)	12.0	4.0	8.0	1765 (C=O), 1575 (C=C), 1185–1205 (C–O–C)
Vb[a]	2.30	2.50	3.45–3.65	4.20	3.55 s, 3.62 s	6.80–7.20 m (4H)	12.0	4.0	8.0	1765 (C=O), 1580 (C=C), 1190–1205 (C–O–C)
Vc[a]	2.35	2.55	3.45–3.65	4.30	3.55 s, 3.62 s	6.50–6.90 m (4H)	12.0	4.0	8.0	1770 (C=O), 1580 (C=C), 1190–1205 (C–O–C)
Vd	2.40	2.55	3.45–3.65	4.20	3.60 s, 3.65 s	6.70–7.10 m (4H)	12.0	4.0	8.0	1770 (C=O), 1580 (C=C), 1185–1210 (C–O–C)
Ve	2.30	2.55	3.40–3.60	4.25	3.60 s, 3.65 s	6.60–7.40 m (4H)	12.0	4.0	8.0	1770 (C=O), 1585 (C=C), 1180–1210 (C–O–C)
Vf	2.15	2.25	3.05–3.40	4.30	3.15 s	6.60–7.70 m (4H)	12.0	4.0	8.0	1775 (C=O), 1580 (C=C), 1195–1205 (C–O–C), 1390 and 1540 (NO_2)

[a] Chemical shifts of the aromatic CH_3 and OCH_3 protons, δ, ppm (s, 3H): 2.30 (**IVb**), 2.35 (**Vb**) 3.70 (**IVa**), 3.77 (**Va**).

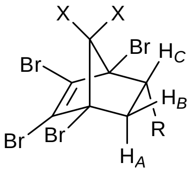
of the transition state is contributed mainly by the symmetric arrangement of the HOMO and LUMO of dienes **I** and **II** and dienophiles **IIIa**–**IIIf**.

By heating mixtures of diene **I** and compounds **IIIa**–**IIIf** in chlorobenzene (12 h, 125°C) we obtained the corresponding adducts **IVa**–**IVf** in high yeids. Diene **II** reacted with compounds **IIIa**–**IIIf** much more readily (6 h, 110°C), leading to formation of adducts **Va**–**Vf**. Compounds **IVa**–**IVf** and **Va**–**Vf** were also prepared by independent syntheses according to Scheme 2. These transformations indicate that the products belong to a single stereochemical series. The structure of compounds **IVa**–**IVf** and **Va**–**Vf** was unambiguously confirmed by their elemental compositions and IR and ^{1}H NMR spectra (Tables 3, 4).

The IR spectra of compounds **IVa**–**IVf**, **Va**–**Vf**, and **VI**–**IX** are characterized by somewhat reduced ν(C=C) frequency (1570–1580 cm^{-1}) due to the presence of bromine atoms at the double bond [2, 3, 18]. The spectra contain strong absorption bands in the regions 1720–1780 [ν(C=O)] and 1130–1210 cm^{-1} [ν(C–O–C)]. Stretching vibrations of the hydroxy group in compounds **VI** and **VII** give rise to a medium-intensity band at 3460 cm^{-1}.

Table 4 contains the ^{1}H NMR parameters of compounds **IVa**–**IVf** and **Va**–**Vf**. Their configuration was determined on the basis of the H_A, H_B, and H_C signals, which form a typical three-spin *ABC* system appearing in the spectrum as three quartets. The geminal coupling constants have their usual values (^2J = 12–12.5 Hz), while the vicinal coupling constants are equal to 4–5 (J_{AC}) and 8–9 Hz (J_{BC}). The latter values slightly differ from those calculated by the Karplus equation, for they depend not only on the dihedral angle but also on the nature of substituents at the same carbon atoms [19, 20]. The identical chemical shifts of H_A, H_B, and H_C suggest similar orientations of the substituent in adducts **IV**–**IX** for the whole series.

It is known that in the ^{1}H NMR spectra of bicyclo[2.2.1]hept-2-enes and their polyhalogenated analogs, signals from the *exo* protons (H_B) appear in a weaker field than those of the *endo* protons (H_A) [20, 21]. Therefore, the coupling constants J_{AC} and J_{BC} correspond to *endo–exo* ($\varphi \approx 120°$) and *exo–endo* interactions ($\varphi \approx 0°$).



Taking into account the chemical shifts of analogous protons in structurally related compounds [2, 3, 5, 18, 22], adducts **IVa**–**IVf**, **Va**–**Vf**, and **VI**–**IX** were assumed to have *endo* configuration.

EXPERIMENTAL

The IR spectra were recorded on a Specord 75-IR spectrophotometer from samples dispersed in mineral oil. The ^{1}H NMR spectra were measured on Tesla BS-467C (80 MHz) and Varian T60 (60 MHz) instruments from solutions in acetone-d_6 using tetramethylsilane as internal reference. The purity of the products was checked by TLC on Silufol UV-366 plates and KSK silica gel (100–150 mesh) in the system benzene–dichloroethane–acetic acid (2 : 4 : 1); spots were visualized by UV irradiation.

Polarographic reduction was performed with the aid of a GWR-673 polarograph in a three-electrode cell at a dropping mercury electrode in DMF using tetraethylammonium iodide as supporting electrolyte; a saturated calomel electrode was used as reference. The second-order rate constants were determined with an accuracy of ±5% from variation of the concentration of dienes **I** and **II**, which was measured by polarography on an ON-101 polarograph [23]. The values given in Table 2 were calculated as average from 3–4 parallel runs. Freshly prepared samples of dienes **I** and **II** and dienophiles **IIIa**–**IIIf** [24] were used in kinetic experiments. The Diels–Alder reactions were carried out in ampules with equimolar amounts of the reactants in chlorobenzene. The reaction mixtures were maintained at a constant temperature within ±0.1°C using an I-10 thermostat. The yields, melting points, R_f values, elemental analyses, and spectral parameters of the obtained compounds are given in Tables 3 and 4.

Reaction of dienes I and II with *para*-substituted allyl benzoates IIIa–IIIf. *a.* Allyl benzoate **IIIa**–**IIIf**, 0.03 mol, was added to a solution of 0.02 mol of diene **I** or **II** in 30 ml of chlorobenzene, and the mixture was heated for 12 h at 125°C (in the reactions with diene **II**, for 6 h at 115°C). When the reaction was complete, the solvent was distilled off under reduced pressure, and adduct **IVa**–**IVf** or **Va**–**Vf** was recrystallized from diethyl ether–hexane.

b. A solution of 0.015 mol of the corresponding *para*-substituted benzoyl chloride in 30 ml of benzene was gradually added to a mixture of 0.01 mol of alcohol **VI** or **VII**, 50 ml of benzene, and 10 ml of pyridine. The mixture was stirred for 3 h at 40–45°C (on a water bath) and washed with water, and the organic layer was dried over Na_2SO_4 and evaporated. Adducts **IVa**–**IVf** and **Va**–**Vf** were recrystallized from diethyl ether–hexane.

c. para-Substituted benzoic acid sodium salt, 0.01 mol, was added to a mixture of 0.015 mol of adduct **VIIIa**, **VIIIb**, **IXa**, or **IXb** and 50 ml of xylene, and the mixture was stirred for 12 h at 125–130°C. When the reaction was complete, the mixture was treated with water and extracted with diethyl ether. The extract was dried and evaporated, and the residue (adduct **IVa**–**IVf** or **Va**–**Vf**) was recrystallized from diethyl ether–hexane. The physical constants of the products obtained as described in *a*, *b*, and *c* were identical.

Reaction of dienes I and II with allyl alcohol. Allyl alcohol, 0.03 mol, was added to a solution of 0.02 mol of diene **I** or **II** in 30 ml of chlorobenzene, and the mixture was heated for 10 h at 120°C (with diene **I**) or for 5 h at 110°C (with diene **II**). The solvent was distilled off under reduced pressure, and adduct **VI** or **VII** was recrystallized from benzene–hexane.

Reaction of dienes I and II with allyl chloride. Allyl chloride, 0.03 mol, was added to a solution of 0.02 mol of diene **I** or **II** in 30 ml of chlorobenzene, and the mixture was heated for 12 h at 125°C (with diene **I**) or for 10 h at 115°C (with diene **II**). The solvent was distilled off under reduced pressure, and adduct **VIIIa** or **IXa** was recrystallized from benzene–hexane.

Following a similar procedure, adducts **VIIIb** and **IXb** were synthesized from dienes **I** and **II** and allyl bromide.

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