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Chemistry of Amine-Boranes. VIII.[1] Reduction of Aldehydes and Ketones with Pyridine-Borane in Trifluoroacetic Acid

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Reduction of aldehydes and ketones with pyridine-borane in trifluoroacetic acid was examined. Symmetric ethers were obtained by the reduction of aldehydes; the combination of an aldehyde and alcohol resulted in the formation of an unsymmetric ether under the same reduction conditions. Aryl ketones and aryl alcohols were reduced directly to the corresponding arenes, while dialkyl ketones were reduced to the alcohols. Alkyl alcohols were not reduced and were recovered intact.

Keywords——pyridine-borane; trifluoroacetic acid; aldehyde; ketone; reduction; ether formation; arene

We have been investigating the reducing ability of pyridine-borane in acids, and have found that pyridine-borane reduces the indole double bond selectively without affecting other functional groups (amide, nitrile, or ester).[3] This method was successfully applied for the modification and determination of tryptophyl groups in peptides.[4] On the other hand, aldehydes and ketones, which are the carbonyl groups most reducible with hydride reagents, have not been subjected to this reduction under the same conditions, because we expected that they would be reduced to alcohols in trifluoroacetic acid, as is the case in refluxing benzene or toluene.[5]

In the present work, we examined the reduction of aldehydes and ketones with pyridine-borane in trifluoroacetic acid and obtained the unexpected results that symmetric ethers were formed by the reduction of aldehydes, while aryl ketones and aryl alcohols were directly reduced to alkylbenzenes. These new reactions were investigated in detail.

Reduction of Aldehydes

It is generally accepted that aldehydes are easily reduced to the corresponding alcohols by borane and borohydride reagents. We investigated the reduction of aldehydes with pyridine-borane in trifluoroacetic acid and found that the products were not the alcohols, but symmetric ethers. These results are presented in Table I.

$$2\mathrm{RCHO}\ \xrightarrow[\text{2) hydrolysis (H}^+\text{ or OH}^-\text{)}]{\text{1) Py}\cdot\text{BH}_3\text{ in CF}_3\text{COOH, 5 min}}\ \mathrm{RCH_2OCH_2R}$$

Chart 1

The procedure is very simple and 5—10 min stirring of the reaction mixture is adequate for the reduction. After the reaction, trifluoroacetic acid was evaporated off *in vacuo*. The infrared (IR) spectrum of the residue showed a strong absorption band at 1780 cm^{-1} due to the trifluoroacetyl group bonded to boron. It is clear that this bond must be formed at the first stage of the reaction. The substitution of the electron-attracting trifluoroacetyl group

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[5] R.P. Barnes, J.H. Graham, and M.D. Taylor, *J. Org. Chem.*, 23, 1561 (1958).

Table I. Synthesis of Symmetric Ethers from Aldehydes

Aldehyde	Yield(%)of ether[a]		MS (m/e, M+)	bp, °C/torr or mp, °C	Reference bp, °C/torr or mp, °C
	10% HCl hydrolysis	10% NaOH hydrolysis			
m-$CH_3OC_6H_4CHO$	63[b]	55[b]	258	175/5	—
C_6H_5CHO	67	87	[c]	128—130/3	295—298/760[d]
p-ClC_6H_4CHO	63(9)	67(treace)	266	54—56(69—70)[e]	54—55[f] (75)[e,g]
p-BrC_6H_4CHO	59(31)	58(39)	354	84.5—85.5(77—78)[e]	85—86[f] (76—76.5)[e,g]
m-$NO_2C_6H_4CHO$	—(56)	—	—	[h]	—
$C_6H_5CH_2CH_2CHO$	72	81	254	168—170/4	220—222/19[i]
$C_{17}H_{15}CHO$	59	70	[c]	131—134/6	288/778[j]

a) Figures in parentheses show the yield (%) of the corresponding alcohols.
b) Ten min reflux for hydrolysis.
c) The corresponding ether was identified by comparison of the IR spectrum and retention time in gas chromatography with those of an authentic sample because no parent ion could be observed in mass spectral analysis.
d) C.W. Lowe, *Ann.*, **241**, 374 (1887).
e) Melting point of the corresponding alcohol.
f) G. Errera, *Gazz. Chim. Ital.*, **18**, 236 (1888).
g) W.H. Carothers and R. Adams, *J. Am. Chem. Soc.*, **46**, 1681 (1924).
h) m-Nitrobenzyl alcohol was identified by comparison of its spectral data with those of an authentic sample.
i) M. Agejewa, *Chem. Zentralbl.*, **76**, **II**, 1017 (1905).
j) A.I. Vogel, *J. Chem. Soc.*, **1948**, 620.

onto the boron atom may stabilize the boron-alkoxyl bond,[6] allowing further reaction to give ethers, as indicated by the absence of the corresponding alcohols. Reduction of m-nitro-, p-bromo-, and p-chloro-benzaldehydes, which have electron-withdrawing groups on the benzene ring, with pyridine-borane resulted in increased yields of the normal reduction product, alcohols. Trifluoroacetic acid is essential for this reduction, because the normal product (alcohols) was obtained when acetic acid was used in place of trifluoroacetic acid.

As the hydrogen atoms of pyridine-borane available for reduction are not consumed completely during reduction in trifluoroacetic acid, it is necessary to decompose them by acid or alkaline hydrolysis before separation of the products. Although the presence of a substantial amount of ethers can be detected before hydrolysis, a small amount of boron complex remains in the acid hydrolysate and is resistant to more concentrated acid hydrolysis, while no boron complex remains after alkaline hydrolysis. The structures of the reaction intermediate and the boron complex that is resistant to acid hydrolysis are still unclear, but the mechanism of ether formation should be similar to that described for the formation of α-chloroethers from boron trichloride and aldehydes.[7]

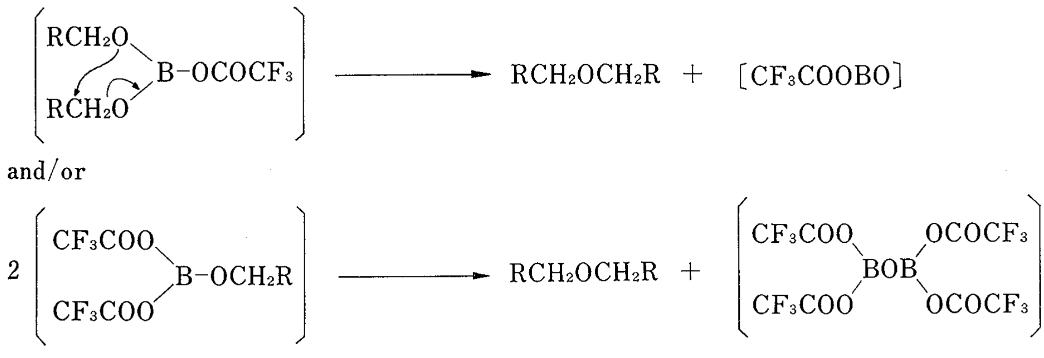

Chart 2

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[6] C.S. Rondestvedt, Jr., R.M. Scribner, and C.E. Wulfman, *J. Org. Chem.*, **20**, 9 (1955).

[7] *a*) M.J. Frazer, W. Gerrard, and M.F. Lappert, *J. Chem. Soc.*, **1957**, 739; *b*) D.K. Black and S.R. Landor, *ibid.*, **1965**, 5225; *c*) W. Gerrard, "The Organic Chemistry of Boron," Academic Press, London and New York, 1961, p. 56.

Table II. Synthesis of Unsymmetric Ethers from Aldehydes and Alcohols

$$\mathrm{R_1CHO + R_2CH_2OH \xrightarrow{Py \cdot BH_3 \text{ in } CF_3COOH} \underset{\mathbf{1}}{R_1CH_2OCH_2R_2} + \underset{\mathbf{2}}{(R_1CH_2)_2O} + \underset{\mathbf{3}}{(R_2CH_2)_2O}}$$

R_1	R_2	**1**		bp, °C/torr	**2**	**3**
		Yield[a] (%)	MS (m/e, M^+)		Yield[a] (%)	Yield[a] (%)
$C_6H_5CH_2CH_2$–	C_6H_5–	63	226	146—148/6	7	6
$C_6H_5CH_2CH_2$–	$CH_3(CH_2)_5CH_2$–	71	248	129—130/1.5	5	Trace
p-ClC_6H_4–	$CH_3(CH_2)_5CH_2$–	82	254	134/2	1	—
p-ClC_6H_4–	C_6H_5–	71	232	144—146/2	5	1

a) Yields were calculated from the gas chromatographic analysis results.

For the synthesis of unsymmetric ethers, the combination of an aldehyde (1 mmol) with an alcohol (2—3 mmol) can be employed. Some results are presented in Table II. Usually small amounts of symmetric ethers (by-products) were formed in addition to the desired unsymmetric ethers, as shown in Table II. In the formation of benzyl phenylpropyl ether by the reduction of phenylpropionaldehyde and benzyl alcohol, a small amount of benzyl ether was detected in gas chromatographic analysis. In a control experiment, benzyl alcohol alone was subjected to the same reduction conditions (alkaline hydrolysis), but it was recovered in 78% yield without isolation of benzyl ether.

Although a useful method for the synthesis of ethers from aldehydes with trialkylsilanes in trifluoroacetic acid has been reported,[8] it can be difficult to separate ethers from silane products when the ethers have boiling points close to those of the silane products (silanols, trifluoroacetate, and disiloxanes),[8b] and by-product formation was often observed. According to a report on reduction with silanes published by a Russian group,[9] the introduction of electron-donating substituents into the aromatic ring permits the hydrogenation of an aldehyde to a methyl group and, in the case of the reduction of aliphatic aldehydes, ether formation competed with alcohol formation (nearly equal amounts). Thus, pyridine-borane reduction seems an attractive alternative method for the preparation of ethers.

Reduction of Ketones

Reduction of the carbonyl groups of aldehydes and ketones to methylenes has been extensively applied in organic synthesis. Of the reductive methods that have been employed, the Clemmensen[10] and Wolff-Kishner[11] reactions have been the most useful. Gribble *et al.* reported the reduction of diaryl ketones[12] and diarylmethanols[13] with sodium borohydride in trifluoroacetic acid to give diarylmethanes in both cases. As described above, the reduction of aldehydes with pyridine-borane in trifluoroacetic acid gave symmetric ethers, and we examined the reduction of ketones under similar conditions. The reduction proceeded smoothly. The products were not ethers,[14] but the corresponding methylene compounds

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8) *a*) M.P. Doyle, D.J. DeBruyn, and D.A. Kooistra, *J. Am. Chem. Soc.*, **94**, 3596 (1972); *b*) C.T. West, S.T. Donnelly, D.A. Kooistra, and M.P. Doyle, *J. Org. Chem.*, **38**, 2675 (1973); *c*) M.P. Doyle, D.J. DeBruyn, S.T. Donnelly, D.A. Kooistra, A.A. Odubela, C.T. West, and S.M. Zonnebelt, *J. Org. Chem.*, **39**, 2740 (1974).

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10) E.L. Martin, *Org. React.*, **1**, 155 (1942).

11) D. Todd, *Org. React.*, **4**, 378 (1948).

12) G.W. Gribble, W.J. Kelly, and S.E. Emery, *Synthesis*, **1978**, 963.

13) G.W. Gribble and R.M. Leese, *Synthesis*, **1977**, 172.

14) In our case no ethers were detected in the reaction mixture, but it has been reported[8c] that some ketones were converted to symmetric ethers by treatment with triethylsilane in trifluoroacetic acid.

when the starting compound had at least one aromatic ring adjacent to the carbonyl group, and the corresponding alcohols were obtained by the reduction of dialkyl ketones. These results are summarized in Table III.

$$\mathrm{ArCOR} \xrightarrow{\mathrm{Py \cdot BH_3\ in\ CF_3COOH}} \mathrm{ArCH_2R}$$

$$\mathrm{(Alkyl)_2CO} \xrightarrow{\mathrm{Py \cdot BH_3\ in\ CF_3COOH}} \mathrm{(Alkyl)_2CHOH}$$

Chart 3

TABLE III. Reduction of Ketones with Pyridine-borane in Trifluoroacetic Acid

Ketone	Reaction conditions[a]	Product	MS (m/e, M+)	Yield (%)	bp, °C/torr or mp, °C	Reference bp, °C/torr or mp, °C
$C_6H_5COC_6H_5$	A	$C_6H_5CH_2C_6H_5$	168	97	112—114/7	60—62/0.35[b]
Anthrone	C	9,10-Dihydroanthracene	180	97	109—110	108—110[b]
9-Fluorenone	A	Fluorene	166	7	110—112	114—114.5[c]
		Bifluorene	330	44	229—230	
		Terfluorene	494	45	277—278	
$C_6H_5CO(CH_2)_3CH_3$	C	$C_6H_5(CH_2)_4CH_3$	148	80	94—95/22	87/12[d]
$C_6H_5COCH_2C_6H_5$	A	$C_6H_5CH_2CH_2C_6H_5$	182	47	50—51	52.5—53[e]
		$C_6H_5CH_2CH(OH)C_6H_5$	198	46	66	67[f]
$C_6H_5COCH_2C_6H_5$	B	$C_6H_5CH_2CH_2C_6H_5$	182	73	50—51	
2-Acetonaphthone	C	2-Ethylnaphthalene	156	81	95—97/5	112.5/12[g]
1-Tetralone	B	Tetralin	132	78	92—94/25	86.5—87.5/17[h]
$C_6H_5CH_2CH_2COCH_3$	A	$C_6H_5(CH_2)_2CH(OH)CH_3$	150	93	104/8	105/6[i]
$C_6H_5CH_2CH_2COCH_3$	B	$C_6H_5(CH_2)_2CH(OH)CH_3$	150	86	104/8	
$C_6H_5CH_2COCH_2C_6H_5$	A	$C_6H_5CH_2CH(OH)CH_2C_6H_5$	212	99	158—160/6	152—154/3.5[j]

[a] A=5—15 min with cooling; B=reflux for 1 hr; C=overnight at room temperature.
[b] G.W. Gribble, W.J. Kelly, and S.E. Emery, *Synthesis*, **1978**, 763.
[c] Reference 21.
[d] A.W. Schmidt, G. Hopp, and V. Schoeller, *Ber.*, **72**, 1893 (1939).
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[f] Reference 16.
[g] H. Luther and G. Wachter, *Chem. Ber.*, **92**, 161 (1949).
[h] H. Adkins and W.A. Reid, *J. Am. Chem. Soc.*, **63**, 741 (1941).
[i] I.E. Muskat and M. Herrman, *J. Am. Chem. Soc.*, **53**, 260 (1931).
[j] Reference 20.

When fluorenone was subjected to this reduction, three products, identified as fluorene, bifluorene, and terfluorene as described in "Experimental", were isolated by silica gel column chromatography with chloroform–hexane (1: 4) as an eluant. This result was considerably different from the reduction of anthrone and other aryl ketones, probably due to the enhancement of reactivity of a fluorene carbocation due to steric factors. Trifluoroacetic acid alone or dilute trifluoroacetic acid (50% $CF_3COOH–CH_2Cl_2$) was effective as a solvent for this reduction, but no alkyl- or aralkyl-benzenes (reduction products) were obtained when dichloromethane or acetic acid alone was used in place of trifluoroacetic acid.

1,2-Diphenylethanol was obtained in 46% yield in addition to bibenzyl (yield, 47%) by the reduction of benzyl phenyl ketone on quenching the reaction mixture during the reduction. When the isolated alcohol was subjected to the same reduction for a longer time (overnight at room temperature or under reflux for 1 hr), bibenzyl was obtained in a good yield. This suggests that aryl alcohols are also reducible to methylenes with pyridine-borane and that the reaction involves the formation of a carbocation which is more stable in the case of aryl compounds with subsequent quenching of the intermediate carbocation by a hydride. Usually pyridine-borane is considered to be a weak reducing agent, and it reduces aldehydes and ketones with difficulty to alcohols in refluxing benzene or toluene.[5] It is interesting

Table IV. Reduction of Benzophenone in Various Solvents

$$\underset{\text{(3 mmol)}}{C_6H_5COC_6H_5} \xrightarrow{Py\cdot BH_3} \underset{\mathbf{4}}{C_6H_5CH_2C_6H_5} + \underset{\mathbf{5}}{C_6H_5CH(OH)C_6H_5} + \underset{\mathbf{6}}{[(C_6H_5)_2CH]_2O}$$

Solvent		$Py\cdot BH_3$ ml	Reaction conditions[a]	Yield (%)		
CF_3COOH, ml	Others (ml)			**4**	**5**	**6**[b]
5	—	0.5	A	97	—	—
2	CH_2Cl_2 (2)	0.6	B	97	—	—
2	THF (2)	0.6	B	97	—	—
0.8	CH_2Cl_2 (3)	0.6	B	28	45	28
0.3	CH_2Cl_2 (4)	0.6	B	—	84	11
—	CH_3COOH (4)	0.6	C	—	95	—
—	CH_2Cl_2 (4)	0.6	C	—	18(77)[c]	—

a) A=5 min with cooling; B=30 min with cooling; C=overnight at room temperature.
b) The structure of **6** was elucidated as bis(diphenylmethyl) ether based on the following data: mp 109—110° recrystallized from hexane (reported[d] mp 109—110°); NMR ($CDCl_3$) δ: 5.40 (2H, s, $>$CH–O–CH$<$), 7.13—7.57 (20H, m, arom H); IR ν_{max}^{Nujol} cm^{-1}: 1055 (C–O–C).
c) Recovery of the starting compound.
d) H. Burton and G.W.H. Cheeseman, *J. Chem. Soc.*, **1953**, 986.

that the reducing power of pyridine-borane in trifluoroacetic acid becomes considerably stronger than in usual organic solvents. As the procedure is very simple and pyridine-borane can be conveniently synthesized[15] and is easy to handle, this method should be useful for the synthesis of alkylbenzenes from acylbenzenes.

Table V. Reduction of Alcohols with Pyridine-borane in Trifluoroacetic Acid

Alcohol	Reaction conditions[a]	Product	Yield (%)
$C_6H_5CH(OH)C_6H_5$	A	$C_6H_5CH_2C_6H_5$	97
$C_6H_5CH(OH)CH_2C_6H_5$	A	$C_6H_5(CH_2)_2C_6H_5$	88
$C_6H_5CH(OH)CH_2C_6H_5$	B	$C_6H_5(CH_2)_2C_6H_5$	55
$C_6H_5CH(OH)(CH_2)_3CH_3$	A	$C_6H_5(CH_2)_4CH_3$	77
2-Naphtyl-CH(OH)CH_3	A	2-Ethylnaphthalene	84
1-Tetralol	A	Tetraline	47
1-Tetralol	B	Tetraline	43
$C_6H_5CH_2CH(OH)CH_2C_6H_5$	A	—	(80)[b]

a) A=overnight at room temperature; B=reflux for 1 hr.
b) Recovery of the starting compound.

Reduction of Alcohols

Several alcohols were subjected to reduction with pyridine-borane in trifluoroacetic acid under reaction conditions similar to those used for the reduction of ketones; the results were summarized in Table V. Aryl alcohols were easily reduced to the corresponding arenes, whereas alkyl alcohols were not reduced and the starting materials were recovered as expected.

Experimental

All melting points and boiling points are uncorrected. The following instruments were used to obtain physical data. Infrared (IR) spectra, Shimadzu IR-400; nuclear magnetic resonance (NMR) spectra (tetramethylsilane as an internal standard), JNM-C-60HL: gas chromatography, Shimadzu GC-4BM. Column

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[15] M.D. Taylor, L.R. Grant, and C.A. Sands, *J. Am. Chem. Soc.*, **77**, 1506 (1955).

chromatography was carried out on silica gel (Merck, Art. 7734) using benzene–hexane (1: 4) or benzene for the separation of ethers and arenes, and benzene–acetone (19: 1) for alcohols.

Materials——Aldehydes, ketones, and benzhydrol were purchased from Tokyo Kasei Kogyo Co. Other starting alcohols were prepared by reduction of the corresponding ketones with $NaBH_4$ in EtOH and were distilled or recrystallized before use. 1,2-Diphenylethanol, mp 66° (reported[16] mp 67°); 1-phenyl-1-pentanol, bp 113—114°/8 torr (reported[17] bp 137°/21 torr); 1-(2-naphthyl)ethanol, mp 74—75.5° (reported[18] mp 71—72°); 1-tetralol, bp 113—114°/5 torr (reported[19] bp 132—134°/12 torr); 1,3-diphenyl-2-propanol, bp 162—164°/6 torr (reported[20] bp 152—154°/3.5 torr). Trifluoroacetic acid was purchased from Wako Chemical Industries Ltd., Tokyo, and used without further purification.

General Procedure for the Reduction of Aldehydes to Symmetric Ethers——Pyridine-borane (0.3—0.4 ml) was added with cooling to a mixture of trifluoroacetic acid (3 ml) and an aldehyde (2.5 mmol). After stirring for 5 min, trifluoroacetic acid was evaporated off *in vacuo*, and 10% HCl or 10% NaOH (15 ml) was added to the residue. The aqueous solution was refluxed for 0.5—1 hr to decompose the boron complex and extracted with benzene (50—60 ml). The benzene solution was dried over anhyd. Na_2SO_4. After removal of the benzene, the residue was purified by column chromatography (SiO_2, benzene for elution) to give an ether, which was distilled and identified by IR and mass spectral analysis.

General Procedure for the Reduction of an Aldehyde and Alcohol to an Unsymmetric Ether——A mixture of an aldehyde (1 mmol), an alcohol (2—3 mmol), and trifluoroacetic acid (3 ml) was treated with pyridine-borane (0.35 ml), and the procedure described above was carried out to give an unsymmetric ether, as shown in Table II. A small amount of symmetric ether (by-product) was also detected by gas chromatographic analysis [the temperatures of the glass column (1.5 m × 3 mm, i.d.), packed with 10% SE-30 on Chromosorb W (60—80 mesh), and the injection chamber were kept at 170° and 200°, respectively]; distillation of the product failed to eliminate the by-product completely.

Typical Procedure for the Reduction of Ketones to Methylenes——Pyridine-borane (0.6 ml, *ca.* 6 mmol) was added to a mixture of 1-tetralone (438.4 mg, 3 mmol) and trifluoroacetic acid (4 ml) with cooling, and the reaction mixture was refluxed for 1 hr. After removal of the solvent *in vacuo*, 10% NaOH (10 ml) was added to the residue and the aqueous solution was heated to 120° (oil bath temperature) for 30 min. The cooled solution was extracted with benzene (30 ml × 2) and the extract was washed with dil. HCl and sat. NaCl, then dried over anhyd. Na_2SO_4. After the removal of benzene, the residue (337.8 mg) was purified by column chromatography [SiO_2, 10 g; benzene–hexane (1: 4) for elution] to give tetralin (308.6 mg), bp 92—94°/25 torr. Yield 78%.

Reduction of 9-Fluorenone——Pyridine-borane (0.6 ml, *ca.* 6 mmol) was added to a mixture of 9-fluorenone (360.3 mg, 2 mmol) and trifluoroacetic acid (5 ml) with cooling, and the reaction mixture was stirred for 15 min. After removal of the solvent *in vacuo*, 10% NaOH (10 ml) was added to the residue and the aqueous solution was heated to 125° for 30 min. The cooled solution was extracted with benzene (30 ml × 2), then the benzene layer was washed with sat. NaCl and dried over anhyd. $MgSO_4$. After the removal of benzene, the residue (542.0 mg) was purified by column chromatography [SiO_2, $CHCl_3$–hexane (1: 4) for elution]. After careful chromatographic purification, three products were isolated and their structures were found to be as follows. Fluorene (7%), mp 110—112°, recrystallized from EtOH (reported[21] mp 114—114.5°). MS *m/e*: 166 (M^+). Bifluorene (44%), mp 229—230°, recrystallized from benzene. MS *m/e*: 330 (M^+); NMR ($CDCl_3$) δ: 3.77 (2H, s, $Ar_2C\langle{}^{H}_{H}$), 5.10 (1H, s, Ar_3C–H), 7.03—7.90 (15H, br. m, arom H). Terfluorene (45%), mp 277—278°, recrystallized from $CHCl_3$. MS *m/e*: 494 (M^+); NMR ($CDCl_3$) δ: 3.68 (2H, s, $Ar_2\overset{|}{C}$–H), 5.08 (2H, s, Ar_3C–H), 7.03—7.09 (22H, br. m, arom H). The precise structures of bifluorene and terfluorene are still unknown, but their NMR spectra show that the 9 position of fluorene is linked to a position on the benzene ring of another fluorene molecule.

General Procedure for the Reduction of Alcohols to Methylene Compounds——The procedure described above was carried out and the corresponding methylene compounds were obtained, as shown in Table V.

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