PHASE TRANSFER CATALYSIS AND HOMOGENEOUS REACTIONS WITH β-OXYALKYL RADICALS FROM ORGANOMERCURIALS

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Abstract— β -Alkoxy- and β -hydroxyalkyl radicals generated by sodium borohydride reduction of oxymercurials react with electron deficient olefins to give addition compounds in an homogeneous process. Heterogeneous reactions are also successful in the presence of catalytic amounts of surfactants and provide a superior method for the "one pot" reductive alkylation of oxymercurials.

The oxy- and aminomercuration-demercuration sequence has been used to prepare the Markovnikov oxy- and amino- compounds from a wide variety of olefins. It involves a simple procedure, easily applied to a large number of structural types¹ (eqn 1).



Sodium borohydride is by far the most popular reagent used for the reduction of β -alkoxy- and β -aminosubstituted organomercurials.² A consistent mechanism for the reduction or organomercurials appears to involve radical intermediates.³ Trapping experiments ^{1a,3b} and rearrangement of unsaturated radicals^{3c} have provided the strongest evidence for radical intermediates in these processes and extended the scope of the sodium borohydride reduction to the formation of C-O and C-C bonds (eqns 2 and 3).^{3c}

In the last few years Giese et al.⁴ have reported on the formation of C-C bonds between "electron rich" and "electron deficient" alkenes in a similar radical reaction. The method consists of the reduction of organomercurials (5) in the presence of "electron deficient" olefins (6) dissolved in dichloromethane which—leads to the synthesis of mono- and difunctionalized alkanes (7) in a regioselective fashion (eqn 4).

A striking feature of the reductive alkylation method is that the nature of the hydride is conditioned by the structure of the organomercurial (5). Paradoxically, sodium borohydride gives very low yields with β -alkoxysubstituted organomercury(II) compounds (3). In this latter case the reduction step is best carried out^{4b,d} by the seldom used and expensive sodium trimethoxyborohydride.⁵.

On these grounds we felt that the clarification of this unprecedented behavior of sodium borohydride in the reduction of β -alkoxyorganomercurials (3) deserved an investigation. In the present paper we wish to report on our study of the influence of the reaction conditions on the course of the reductive alkylation of β -alkoxy(hydroxy) substituted organomercurials (3) which allows the use of sodium borohydride as a general reducing agent, and the development of a new and very convenient procedure catalyzed by surfactants.

RESULTS AND DISCUSSION

The reduction of organomercurials (5) with sodium borohydryde has been essayed in a number of different



reaction conditions. The best results are obtained by the use of Brown's method,^{2b} that is, when organomercuric salts in strongly alkaline solutions (as the organomercuric hydroxides) and THF added as cosolvent for the mercuration step are reduced by solutions of sodium boro-hydryde in basic media. The merit of these conditions is to get in a single phase the organomercurial and the reducing agent in a solvent in which both give stable solutions. The reduction of compounds 3 goes through the formation of a β -alkoxy-(or hydroxy) alkyl radical which is able to undergo a facile β -elimination process. For this reason it becomes crucial to perform the reduction in a single phase medium and hence the success of Brown's method.

We have attempted to extend this method of reduction to perform the reductive alkylation of β -substituted oxymercurials (3) and found two added difficulties, namely, the competitive process of simple reduction (path a, eqn 5) and the problem of introducing the hydrophobic "electron deficient" alkene (6) into the alkaline aqueous medium. When reactions were carried out with dichloromethane as cosolvent (eqn 5), the reaction products were found to derive almost exclusively from path a, and only trace amounts of the desired compounds 7 could be detected.

The use of other inert solvents with sodium borohydride increases the extent to which path b is followed (Table 1). For instance, a mixture of THF and dichloromethane gives up to 23% yield of 7 (entry 2, Table 1). Similar results are obtained with dioxane (entry 3, Table 1). In DMF as cosolvent, path b predominates and compounds 7 were obtained with yields up to 66% (Table 1). Since the polarity⁶ of 1, 4-dioxane $E_T(30) = 36.0$ or THF $E_T(30) = 37.0$ is smaller than that of dichloromethane $E_T(30) = 41.2$ and DMF $E_T(30) = 43.8$ it is clear that the cosolvent polarity is not by itself a deter-



Table 1. Synthesis of 5-methoxyesters and nitriles 7 (eqn 5)^a

Entry_	R ¹	R ²	1	Y	3	6	7	Cosolvent	Yield &
1	с ₆ н ₅	н	a	сı	a		a	DMF	53
2			a		a	a	a	THF/CH2C12	23
3			a		a	a	a	Dioxane	26
4			a		a	b	ь	DMF	42
5	с ₆ н ₅ сн ₂	้ห	Ъ	Cl	ь	a	с	DMF	55
6			b		b	b	đ	DMF	46
7	^С 6 ^Н 5	^{СН} 3	с	C1	с	a	eÞ	DMF	46
8	-(CH ₂)	3	đ	C1	đ	a	£°	DMF	60
9			đ		đ	b	a _c	DMP	54
10	-(CH ₂)4	-	e	Br	e	a	hď	DMF	66
11			e		e	b	1d	DMF	51
12	^{n-C} 6 ^H 13	н	f	Br	f	a	t	DMF	57
13 ^e			f		f	a	t	THF	5
14 ^e			a		a	a	a	THF/CH2C12	<5
15 ^e			ь		b	a	с	DMF	19
16 ^e			đ		đ	a	£	THF/CH2C12	6
17 ^e			đ		đ	a	f	CH2C12	<5
18 ^e			e		e	ъ	1	CH2C12	<5
19 ^e			f		f	a	t	DMF	7

^aReactions were carried out at 0°. Elemental mercury was recovered in nearly quantitative amount. ^bEquimolecular mixture of diasteroisomers. ^c > 90% Trans. ^aTrans/cis misture 70/30 ^cWater was substituted for aqueous sodium hydroxide in the general procedure.

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minant factor for the C-C bond formation reaction to take place. On the contrary the progress of the alkylation reaction seems to depend on the ability of the water-cosolvent system to mix the sodium borohydryde, the organomercurial (3) and the "electron deficient" olefin (6) in a single phase.

Since the reduction is an exothermic process an icewater bath is used to avoid overheating. Otherwise, the yield is slightly lowered. A series of runs were performed under neutral conditions by adding sodium borohydride desolved in a small amount of water to a solution of compounds **3** and **6** in different organic solvents (entries 13-19, Table 1). In this case yields were lowered specially when primary radicals were involved. The course of the reaction could be studied at temperatures below 0° in THF/dichloromethane due to the low freezing point of the mixture. At -60° the reduction is very slow and only at about -40° the reaction goes easily to completion. However, under these conditions path a largely predominates.

The usefulness of sodium borohydryde in the reduction of different types of functional groups (i.e. carboxylic acids,⁷ alkyl halides,⁸ etc) has been greatily broadened by the use of quaternary ammonium and phosphonium salts and crown ethers as phase transfercatalysts in aqueous alkaline/organic reaction media. For this reason we felt that PTC should be successful in the reductive alkylation processes. Initially we attempted the reaction of the methoxymercurial derived from 1a dissolved in 10% aqueous sodium hydroxide with sodium borohydryde and a solution of acrylonitrile (6a) in dichloromethane in the presence of hexadecyltrimethylammonium bromide (entry 1, Table 2) and found that it affords 7a in 44% yield. Then, under PTC conditions the participation of path b has been in the overall process nearly as important as in the case of the homogeneous reactions (entry 1, Table 1). Some ammonium salts and other typical phase transfer catalysts give products contaminated by the transference agent and have a rather high cost. For these reasons, we essayed to perform the reductive alkylation in the two phases system by using as the catalyst nonionic surfactants which are cheap and can be eliminated from the organic layer by washing with water. Non-ionic surfactants are composed of a long hydrophobic alkyl group connected to a highly polar neutral group and are readidly absorbed at interfaces and aggregate easily in micelles. Then, these substances should be succeptible to promote reactions between hydrophobic organic moieties in an organic solvent with other reagents dissolved in water.

When a mixture containing catalytic amounts of Triton X-100,¹⁰ the oxymercurial (3) dissolved in 10% aqueous NaOH, and the "electron deficient" olefin (6) in dichloromethane is reduced by a solution of sodium borohydride in aqueous sodium hydroxyde, the product 7 is formed in good yield. Reactions can be performed even more easily in a "one pot" process if the oxymercurial (3) is synthesized *in situ* from the corresponding olefin (1), the nucleophile (2, water or alcohol), and a Hg(II) salt. The reductive alkylation (eqn 6) is then carried out by addition of Triton X-100, alkene (6), and finally sodium borohydride to the oxymercuration mixture (Experimental and Table 2).

When 1 is a terminal monoolefin the reaction is regiospecific. Compounds 7 are obtained as a nearly equimolecular mixture of diasteroisomers in the case of unsymmetrically substituted alkenes (entry 7, Table 1). This mixture was determined by the duplicity of signals displayed in ¹³C- and ¹H-NMR. Cyclic monoolefins (1d, e) give diastereoisomeric mixtures whose composition depends on the ring size. When the oxymercurial (3) derives from cyclohexene (1e) the *trans/cis* isomer ratio found was about 70/30 (by NMR) but in the case of cyclopentene (1d) the product was more than 90 per cent *trans.* The *trans* structure of the major isomer in compounds 71 and 7g was assumed on the basis of the stereochemistry found for cyclohexyl derivatives (7h, i, k).

cis-cis-1, 5-Cyclooctadiene (1g) was mercurated in water with mercury(II) nitrate to afford in a few seconds corresponding dimercurated 1, 5-epoxide (3h). The reductive alkylation by the surfactant method (eqn 7)

Entry_	R ¹	R ²	1_	.R ⁵	x	3	6	7	Yield &
1 ^c	C6H5	н	a	СНЗ	OAC	-	a	a	44
2	C6 ^H 5	н	a	сн3	OAc		a	a	50
3	с ₆ н ₅ сн ₂	н	ь	СНЗ	OAc		a	c	51
4	C6 ^H 5 ^{CH} 2	H	Ъ	СН3	OAc		ь	d	39
5	-(CH ₂) ₄ -		е	СНЗ	OAc		Ь	ĩq	60
6	-(CH ₂) ₄ -		e	н	OAC	g	a	k d	59
7	-(CH ₂) ₂ -CH=CH-(CH ₂) ₂	-	g	н	NO3	h	ь	1 d	61
8	CH2=CH-(CH2)2-	н	h	н	OAc	ĩ	ь	»e	40
9	C6 ^H 5 ^{CH} 2	H	ь	н	OAC	ţ	b	D	57
10	n∽C4 ^H 9	H	i	н	OAC	k	Ь	o	48

Table 2. Reductive alkylation catalyzed by surfactants (eqns 6, 7 and 8)^{a,b}

^aReactions were carried out at 0° in dichloromethane solution and Triton X-100¹⁰ as the catalyst. Elemental mercury was recovered in nearly quantitative amount. ^bSimilar results were obtained when isolated mercurials (Table 1) were used instead of the "one pot" procedure. ^cHexadecyltrimethylammonium bromide was substituted for Triton X-100 as the catalyst. ^dTrans/cis misture 70/30 (by ¹H- and ¹³C-NMR). ^cTrans/cis mixture 65/45 (by ¹H- and ¹³C-NMR).



takes place with partial inversion at C-2 and C-6 to afford 71 as a 70/30 of *trans/cis* isomers (by ¹³C-NMR).¹¹ Reactions of 1g and Hg(II) acetate were not taken into account since in this case, the oxymercuration step yields a mixture of dimercurated 1, 4- and 1, 5-epoxides¹² from which a more complex mixture of reductive alkylation compounds would result. A nearly equimolecular mixture of *cis*- and *trans*-7m was obtained from 1, 5-hexadiene (1h). It should be noted that the oxymercuration conditions could modify the isomers¹² ratio since the stereochemistry of the compound 7m is prefixed by that of the oxymercuric adduct 3i.



The reductive alkylation of methyl acrylate (6a) by the surfactant method of hydroxymercurials prepared *in situ* from mono-olefins (entries 9 and 10, Table 2) followed by gentle heating of the crude reaction product allowed the direct isolation of the corresponding lactones in good yields (eqn 8).

tion are the major or even exclusive reaction paths. On the above grounds we believe that the two phases reaction promoted by surfactants described by us provides a superior method for the reductive alkylation of oxymercurials (3) with sodium borohydride in terms of generality, convenience and availability of all reagents used.

EXPERIMENTAL

Solvent extracts of products were appropriately washed and dried (Na₂SO₄) before removal of the solvent. NMR spectra were recorded by using a Varian EM-390 and a Varian FT-80A spectrometers. Chemical shifts are reported in ppm(δ) down field from Me₄Si. For TLC and column separations silica gel (Merck) was used with ether as eluant. Electron deficient olefins were distilled prior to use.

Synthesis of 5-methoxyesters and nitriles (7) in homogeneous basic medium (Entries 1-12, Table 1). To a stirred soln of $3^{13}(10 \text{ mmol})$ in 1N NaOH (10 mL) an excess of 6 (50 mmol) in the corresponding cosolvent (40 mL) (Table 1) was added. This mixture was cooled in an ice-cooled bath and NaBH₄ (10 mmol) dissolved in 1N NaOH (5 mL) was dropwise added. After 30 min quantitative elemental Hg was filtered off and the resulting soln extracted with ether. After usual work up procedure the residue was purified by column chromatography or vacuum distillation. Yields in compounds 7 are summarized in Table 1. Spectral data are collected in Table 3.

Synthesis of 7 in homogeneous neutral medium (entries 13-19). A soln of NaBH₄ (10 mmol) in water (5 mL) was dropwise added to a mixture of 3 (10 mmol) and 6 (50 mmol) in the corresponding solvent (50 mL) (Table 1) at 0°. The procedure above described gives the corresponding compound 7 in poor yield (2-19%, Table 1).



CONCLUSIONS

It is well documented that the success in the sodium borohydride reduction of oxymercurials greatly depends on the selection of the adequate experimental conditions. Among them, the use of a basic homogenous medium appears to be one of the most important facts. This becomes critical in the reductive alkylation reaction since, in this case, it must be overcome the added difficulty of getting the hydrophobic reagent 6 ready to interact with the intermediate β -alkoxy(-hydroxy) substituted radical resulting from the first step of the reduction. Otherwise, the reductive alkylation fails, and the competitive "normal" reduction and/or deoxymercuraAttempt of reductive alkylation of 3a at low temperature. A soln of 3a (3.71 g, 10 mmol) and 6a (50 mmol) in a mixture of THF (90 mL) and CH_2Cl_2 (90 mL) was cooled at -60° . Then, NaBH₄ (0.36 g, 10 mmol) was slowly added. At this temp. the reaction was very slow but when the mixture was warmed to -40° Hg precipitated and was filtered out at room temp. The resulting soln was evaporated and the residue analyzed by NMR. Compound 7a could not be detected.

"One pot" synthesis of 5-methoxyesters and nitriles 7 in heterogeneous medium (entries 1-5, Table 2). A soln of 1 (10 mmol) in anhyd MeOH (20 mL) was treated with mercuric acetate (10 mmol) until negative test for Hg^{2+} with 1N NaOH. The oxymercuration mixture was treated with 1N NaOH (25 mL), Triton X-100 (0.5 g) and 6 (50 mmol) in CH₂Cl₂ (25 mL). The heterogeneous mixture was cooled at 0° with an ice-water

Table 3. Spectral data^a for compounds 7^{b}

Compound		¹³ C-NMR [CC1, 6(TMS)]
Z	1.55 (m,2H), 1.90 (m,2H), 2.25 (t,2H), 3.20 (s,3H), 4.10 (t,1H)	16.02, 21.28, 36.19, 55.66, 82.12, 119.01, 125.89, 127.01,
	7.25 (s, arom.)	127.85, 141.26
7 b	1.65 (m,2H), 1.85 (m,2H), 2.35 (t,2H), 3.20 (s,3H), 2.65 (s,3H)	20.59, 32.81, 36.75, 50.09, 55.35, 82.72, 125.83, 126.68, 127.60
	4.15 (t,1H), 7.25 (s, arom.)	141.71, 172.34
70	1.35-1.85 (m,4H), 2.25 (t,2H), 2.40 (d of d,2H), 3.20-3.50 (m,	16.06, 20.82, 31.64, 39.00, 55.88, 80.43, 119.23, 125.52, 127.66
	1H), 3.30 (s,3H), 7.15 (br. s, arom.)	128.85, 138.12
74	1.20-1.85 (m,4H), 2.20 (d,2H), 2.75 (t,2H5, 3.20-3.40 (m,1H),	18.56, 31.61, 32.57, 38.76, 49.75, 55.34, 80.57, 124.78, 126.96
	3.30 (s,3H), 3.60 (s,3H), 7.15 (s, arom.)	128.19, 137.79, 172.09
Te	0.80 and 0.95 (d,d,3H), 1.25-2.45 (m,4H), 3.15 and 3.20 (s,s,3H),	13.21 and 14.43, 13.94, 27.59, 37.82, 55.61 and 55.90, 85.94 and
	3.80 and 3.95 (d,d,1H), 7.25 (br. s, arom.)	87.06, 124.65 and 124.96, 126.26, 126.65, 127.21, 138.89 and 139.41
trans-7f	1.00-2.10 (m,9H), 2.40 (t,2H), 3.30 (s,3H), 3.75 (m,1H)	15.00, 21.59, 29.10, 29.31, 30.11, 44.08, 55.72, 86.93, 119.32
trans-78	1.45-2.05 (m,9H), 2.40 (t,2H), 3.30 (s,3H), 3.60 (m,1H), 3.7	21.57, 28.53, 28.31, 30.02, 31.83, 44.19, 50.16, 55.48, 87.06,
	(s, 3H)	172.61
trans-7	1.00-3.00 (m,13H), 3.30 (s,3H), 3.75 (m,1H)	14.17, 23.73, 24.76, 28.45, 29.61, 29.61, 41.60, 54.82, 82.56,
c		119.37
cis-7h	1.00-3.00 (m,13H), 3.25 (s,3H), 3.50 (m,1H)	13.85, 20.02, 24.17, 26.22, 26.74, 26.83, 39.19, 54.97, 76.61,
(119.25
trans-71	0.85-3.00 (m,13H), 3.30 (s,3H), 3.65 (s,3H), 3.75 (m,1H)	23.78, 24.76, 27.36, 29.55, 29.70, 30.97, 41.87, 50.05, 54.70,
(82.65, 172.74
cis-7i	0.85-3.00 (m,13H), 3.25 (s,3H), 3.50 (m,1H), 3.60 (s,3H)	20.06, 24.41, 26.17, 26.49, 27.12, 30.80, 39.78, 50.05, 54.94
		77.21, 172.66
ľ,	0.65-1.55 (m,17H), 2.35-2.70 (m,2H), 3.20 (s,3H), 3.80-4.10	13.76, 18.42, 22.33, 25.58, 30.24, 31.59, 38.75, 46.45, 55.14,
	(m, 1H)	65.52, 81.66, 117.04
trans- T	1.00-3.00 (m,13H), 3.10 (br. s,1H), 3.70 (m,1H)	14.07, 23.97, 24.60, 28.12, 29.39, 35.00, 43.16, 73.16, 119.70
cis- n	1.00-3.00 (m,13H), 3.10 (br. s,1H), 3.50 (m,1H)	13.84, 19.68, 23.97, 25.50, 26.57, 32.06, 39.38, 66.84, 119.68
trans-71°		15.93, 22.17, 24.99, 28.94, 29.11, 29.30 ^d , 30.87, 35.67, 50.00
	1.00-2.40 (m,18H), 3.50 (s,6H), 3.50-370 (m,2H)	65.47, 76.93, 171.72
cis-7		20.40, 25.24, 28.04, 30.87 ^d , 35.96, 50.00, 69.39, 171.72
trans-7m	(H3 5) (2 (H) - 2 (H)	18.43, 30.64, 30.64, 32.02, 47.98, 75.55, 170.53
cis-7m°	1 10. 2 0 0 2 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	18.43, 27.73, 28.70, 32.17, 47.98, 74.83, 170.53
7n	1.20-2.50 (m,6H), 2.50-3.00 (m,2H), 4.40 (m,2H), 7.15 (s, arom.)	17.05, 25.84, 28.15, 40.81, 79.66, 125.57, 127.32, 128.42, 135.53
		170.57
70	0.80-2.60 (m,15H), 4.20 (m,1H)	13.54, 18.14, 22.15, 26.73, 27.48, 29,12, 35.20, 80.24, 171.66
	*Nitriles displayed the IR $\nu_{\rm CN}$ absorption at 2250 ± 5 cm $^{+1}$	vco for methylesters and lactones were found at
	1740 \pm 10 cm ⁻¹ . Satisfactory microanalytical values were fou ^c Data are from the cis-trans isomers mixture. ^d Uncertain assign	d for all compounds: $C \pm 0.35$; $H \pm 0.15$; $N \pm 0.10$. nement.

bath and then NaBH₄ (10 mmol) dissolved in 1N NaOH (5 mL) was dropwise added. After 30 min quantitative elemental Hg was filtered out and the resulting soln extracted with CH_2Cl_2 . After usual work up procedure the oil residue was purified by column chromatography.

"One pot" synthesis of compounds 7k-0 (entries 6-10, Table 2). A suspension of 1 (10 mmol) in water (10 mL) was treated with the corresponding Hg(II) salt [10 mmol for monoolefins (entries 6, 9 and 10) or 20 mmol for diolefins (entries 7 and 8)]. The oxymercuration mixture was treated with 2N NaOH (20 mL), Triton X-100 (0.5 g) and 6 (50 or 100 mmol) in CH₂Cl₂ (25 ml. The heterogeneous mixture was cooled at 0° with an ice-water bath, and then NaBH₄ (10 or 20 mmol) dissolved in 1n NaOH (5 mL) was dropwise added. After 30 min quantitative elemental Hg was filtered out and the resulting soln extracted with CH₂Cl₂. Compounds 7k-m were isolated according the usual work up procedure and the oil residue purified by column chromatography. The crude product from entries 9 and 10 (Table 2) was heated at 100-120° in an oil bath and distilled under vacuum to afford lactones 7n and 70.

trans-2, 6-Bis(nitratomercurio)-1, 5-epoxycyclooctane (3h). A suspension of 1g (10 mmol) in water (10 mL) was treated with mercury(II) nitrate monohydrate (20 mmol) and stirred for 5 min to afford 3h. Spectral data (13 C-NMR in NaOHaq) found for 3h were identical to those reported by Bloodworth.¹¹

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