The physical constants of the lactones were as follows: 1, mp 97–99 °C; **1d**, mp 97–100 °C; *Z*-**2d**, mp 134–135 °C; **3**, mp 149–151 °C; **4**, mp 78–80 °C; **5**, bp 145–148 °C (0.3 mm); **6**, mp 69–72 °C; **7**, mp 110–112 °C; **8**, bp 180–185 °C (0.6 mm).

The NMR spectra of the lactones are presented in Table I.

Bromination of α,α-Dimethyl-γ-benzyl-γ-phenyl-γ-butyrolactone. To 2.80 g (0.01 mol) of the title lactone in 50 mL of CCl_4 was added 1.78 g (0.01 mol) of *N*-bromosuccinimide and 50 mg of benzoyl peroxide. The mixture was stirred and irradiated with a sunlamp for 3 h and then cooled. Succinimide was removed by filtration and the filtrate was washed with hot water, dried, and concentrated, yielding a white crystalline solid. Recrystallization from ether–chloroform (1:1 v/v) gave α,α-dimethyl-γ-(α-bromobenzyl)-γ-phenyl-γ-butyrolactone (**3**): 2.99 g (75%); mp 149–151 °C.

Anal. Calcd for $C_{19}H_{19}O_2Br$: C, 63.50; H, 5.29. Found: C, 63.56; H, 5.30.

Deuterium Exchange with (*Z*)-α-Methyl-γ-(α′,α′-dideuteriobenzyl)-γ-phenyl-γ-butyrolactone. (*Z*-2d). A small piece of lithium wire was dissolved in 5 g of D_2O. In a 10-mL flask was placed 0.5 g of the title lactone dissolved in a few milliliters of 1,2-dimethoxyethane. The lithium deuteroxide solution was added and then more 1,2-dimethoxyethane was added dropwise until the mixture became homogeneous. The solution was heated at 80 °C for 1 h and stirred at room temperature overnight. The solvent was evaporated and then D_2O was added, producing a cloudy solution from which white crystals soon separated. The crystals were washed with D_2O and recrystallized from methanol; 0.4 g of product was recovered, mp 134–135 °C. The NMR spectrum of the recovered lactone indicated partial exchange of hydrogen in the α position. The deuterium exchange procedure was repeated, giving the deuterated lactone whose NMR spectrum is described in the body of the paper.

Registry No.—Methyl β-benzoylpropionate, 25333-24-8; methyl α-methyl-β-benzoylpropionate, 36057-38-2; methyl α,α-dimethyl-β-benzoylpropionate, 15118-66-8.

References and Notes

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(3) C.-E. Low and R. M. Roberts, *J. Org. Chem.*, **38**, 1909 (1973).
(4) A. A. Khalaf and R. M. Roberts, *J. Org. Chem.*, **38**, 1388 (1973). Although the *Z,E* assignment of these isomers was tentative at the time this paper was published, a subsequent X-ray crystal structure analysis confirmed the assignment unequivocally as correct.[5]
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New Mild Conditions for the Synthesis of α,β-Unsaturated γ-Lactones. β-(2-Phthalimidoethyl)-$\Delta^{\alpha,\beta}$-butenolide

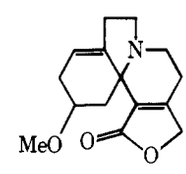
Scott F. Krauser[1] and Arthur C. Watterson, Jr.*

Contribution from the Organic Chemistry Program, Department of Chemistry, University of Lowell, Lowell, Massachusetts 01854

Received February 23, 1978

In connection with a synthetic approach to D ring lactone erythrina alkaloids,[2] particularly cocculolidine (**1**),[3] we needed an efficient route to β-(2-phthalimidoethyl)-$\Delta^{\alpha,\beta}$-butenolide (**2a**).

Despite the variety of methods that have been devised for the synthesis of the frequently encountered $\Delta^{\alpha,\beta}$-butenolide system[4] and the relatively simple structure of this particular example, such a route proved to be surprisingly difficult to develop. We did meet with some initial success in that the reaction between ethyl bromoacetate and 1-acetoxy-4-phthalimido-2-butanone (**3a**) under classic Reformatsky conditions gave[5] acetoxy lactone **4**, which we find eliminates HOAc on heating in *N,N*-diethylaniline to give **2a** cleanly and



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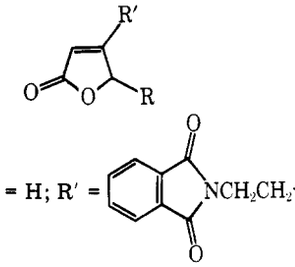
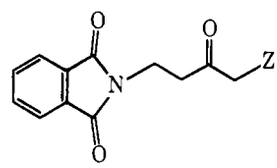


2a, R = H; R′ = NCH_2CH_2-

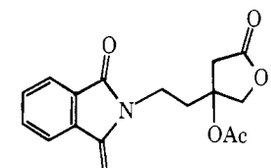
b, R = H; R′ = Ph
c, R = R′ = Ph
d, R = R′ = $-(CH_2)_4-$

in high yield. The Reformatsky reaction with α-acetoxy ketones has recently been stated[4] to be the method of choice for the preparation of $\Delta^{\alpha,\beta}$-butenolides. Unfortunately, in spite of considerable effort, the best yields that could be obtained in that step were low (20–21%) and even then somewhat variable with respect to product isolation, so that a better overall route to **2a** was needed.

More recent variations[6,7] in the Reformatsky procedure as well as other established methods[8–11] of introducing appropriate two carbon units as applied to 1-substituted 4-phthalimido-2-butanones (**3**), with the exception of BF_3-catalyzed addition of ethoxyacetylene[12] to α-chloro ketone **3b**,[5] were useless. The latter reaction gave in about 50% yield a product which appeared, as judged by the NMR spectrum, to be the expected α,β-unsaturated esters **5**, but without selectivity with respect to the required *Z* isomer and in any case as a very dark oil which resisted attempts at purification and complete characterization.



3a, Z = OAc
b, Z = Cl
c, Z = N_3
d, Z = $OCOCH_2Br$
e, Z = Br
f, Z = $OCOCH_2P^+Ph_3\ Br^-$
g, Z = $OCOCH{=}PPh_3$



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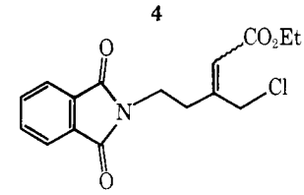

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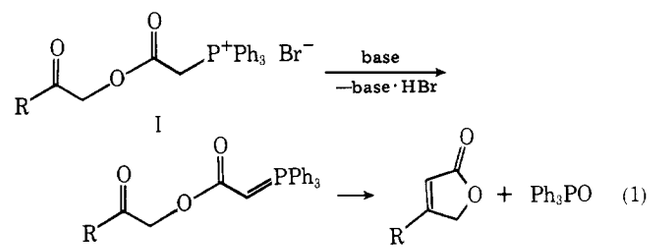
We then turned to an intramolecular approach to carbon–carbon bond formation, specifically via the Wittig reaction (eq 1).



I

(1)

Compounds of type I have been cyclized to $\Delta^{\alpha,\beta}$-butenolides once before. In the two reported[13] examples (eq 1, R = steroidal), similar moderate yields were obtained using the rather diverse systems NaH/Me_2SO (100 °C for 4 h; 51%) and K_2CO_3/*t*-BuOH (reflux 8 h; 67%); apparently, the exact nature of the base is not critical, and thus it seemed that one more compatible with our system could be effective. It was further hoped that less drastic conditions (purification was by chromatography) would suffice.

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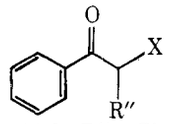
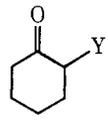
The details of the preparation of type I salts have not been previously published. On heating with bromoacetic acid in C_6H_6, diazo ketone **3c**[5] was converted to bromoacetoxy ketone **3d;** a cleaner alternative was to treat **3c** with aqueous HBr to give bromo ketone **3e** and react this with $Et_3N/BrCH_2CO_2H$ to give **3d.** The latter two reactions proceeded readily at room temperature in Me_2CO, and isolation of **3e** was unnecessary. Treating **3d** with Ph_3P (3 equiv) in C_6H_6 at 25 °C gave the requisite phosphonium salt **3f;** both conversions of **3c** → **3f** were essentially quantitative, as is the preparation[5] from β-alanine of **3c** itself.

When a suspension of **3f** in excess Et_3N was stirred at room temperature overnight, there was obtained in high yield a white solid consisting of Et_3N·HBr and a new air-unstable material. The identity of this compound as phosphorane **3g** was clear from the IR spectrum, which in the 1650–600 cm^{-1} region is very similar to that of ethoxycarbonylmethylenetriphenylphosphorane[14] ($Ph_3P{=}CHCO_2Et$) and marked by an intense band at 1625 cm^{-1} (P=C). That this highly reactive intermediate could be intercepted at all is attributable to its very low solubility in the Et_3N, because mere dissolution in a good solvent, e.g., $CHCl_3$, resulted in its virtually instantaneous and quantitative conversion to **2a** and Ph_3PO.

Both steps could be easily and cleanly carried out in one operation by simply treating the phosphonium salt with a slight excess of Et_3N under homogeneous conditions. Thus, a CH_2Cl_2 solution of **3f** containing 1.2 equiv of the amine allowed to stand at room temperature gave a quantitative yield of the desired product mixture, from which **2a** was obtained by recrystallization in 71% yield. Continuous monitoring of the reaction at ca. 40 °C by IR spectroscopy (see Experimental Section) revealed it to be complete within 30 min at this temperature.



a, R″ = H; b, R″ = Ph
6, X = Br
7, X = $OCOCH_2Br$
8, X = $OCOCH_2P^+Ph_3\ Br^-$



9a, Y = $OCOCH_2Br$
b, Y = $OCOCH_2P^+Ph_3\ Br^-$
c, Y = $OCOCH_2P(O)(OEt)_2$

As this procedure constitutes an especially efficient and mild overall route to a $\Delta^{\alpha,\beta}$-butenolide, its generality was briefly explored; the conversion of **3e** to **3d** had extended the range of potential starting materials from α-diazo ketones to the more generally available α-bromo ketones, and the latter were utilized. The 2-bromoacetophenones **6a** and **6b** reacted smoothly via the respective intermediates **7a,b**[15] and **8a,b**[15] to give the corresponding butenolides **2b**[16] and **2c,**[17] isolated as was **2a.** The cyclizations, which again gave spectrally clean mixtures of lactone, Ph_3PO, and Et_3N·HBr, were monitored as before and found also to proceed rapidly (Table I).

As applied to the preparation of bicyclic lactone **2d,**[18] the same sequence failed in the first step, with little if any bromoacetoxy ketone **9a** resulting from the treatment of 2-bromocyclohexanone[19] with the $Et_3N/BrCH_2CO_2H$ reagent. The preparation of phosphonium salt **9b** was not further explored, but instead a mild intramolecular[13] route to **2d** through the Horner–Emmons[11] reagent **9c** was examined. Reaction of 2-bromocyclohexanone with potassium diethylphosphonoacetate[20] [$(EtO)_2P(O)CH_2CO_2^-K^+$] at room temperature gave **9c**[15] directly in 91% yield, the use of the preformed phosphonate thus avoiding both bromo ester **9a** and the usual high temperature Arbuzov conditions used for the preparation of phosphonates.[13,21] The cyclization to **2d** could be accomplished, but comparably mild conditions were ineffective and the crude product obtained (NaH, refluxing C_6H_6, ca. 50% yield) was admixed with unidentified materials; this route to **2d** is, however, potentially advantageous for its brevity.

Table I. Cyclization of Phosphonium Salts[a]

salt	registry no.	salt concn, M	product[b]	registry no.	reaction time, h[c]
3f	66792-64-1	0.071	**2a**	66792-67-4	0.5
8a	66792-65-2	0.10	**2b**	1575-47-9	0.5
8b	66792-66-3	0.10	**2c**	6620-27-5	0.1

[a] All reactions carried out in $CHCl_3$ at ~40 °C using 1.2 mol of Et_3N/mol of salt. [b] As equimolar mixture with Ph_3PO and Et_3N·HBr. [c] As indicated by IR analysis (see Experimental Section).

Type I phosphonium salts are thus seen to cyclize to give $\Delta^{\alpha,\beta}$-butenolides with a facility not previously appreciated. This factor and the ease with which the salts may be prepared combine to produce a route to these lactones which gives excellent overall yields while completely avoiding elevated temperatures. Comparable phosphonates may also be obtained under equally mild conditions, although their cyclization has not been effected as easily.

Experimental Section

Melting points are uncorrected. IR spectra were obtained with a Beckman 4230 spectrophotometer. NMR spectra were recorded on a Perkin-Elmer R-24 (60 Mz) instrument or a Jeol PS-100 (100 Mz, FT mode) using $CDCl_3$ as solvent and Me_4Si as an internal standard. Combustion analyses were performed on a Perkin-Elmer 240 automatic elemental analyzer.

1-Bromoacetoxy-4-phthalimido-2-butanone (3d). A. Directly from 3c. A solution of 1.5 g (0.0064 mol) of diazo ketone **3c**[5] and 1.1 g (0.0079 mol) of bromoacetic acid in 40 mL of dry benzene was heated at 65–80 °C for 2.5 h, cooled, washed with H_2O and 1 N $NaHCO_3$, dried ($MgSO_4$), and evaporated to give 2.2 g (96%) of **3d** as a yellow solid, mp 106–111 °C. An analytical sample had mp 114.5–115 °C (C_6H_6–cyclohexane); NMR δ 2.9 (t, 2 H, J = 7 Hz), 3.9 (s, 3 H), 4.0 (t, 2 H, J = 7 Hz), 4.8 (s, 2 H), 7.8 (m, 4 H); IR (Nujol) 1760, 1715 cm^{-1}.

Anal. Calcd for $C_{14}H_{12}BrNO_5$: C, 47.48; H, 3.42; N, 3.95. Found: C, 47.48; H, 3.31; N, 3.89.

B. Via 1-Bromo-4-phthalimido-2-butanone (3e). To a solution of 10.54 g (0.0434 mol) of **3c** in 160 mL of Me_2CO was added 48% HBr dropwise until gas was no longer evolved (0.25 h). The solution was stirred another 0.25 h and evaporated to give 12.44 g (97% crude yield) of **3e** as an off-white free-flowing solid. One recrystallization from EtOH gave 10.53 g (82%) as pure white needles, mp 113–114 °C. An analytical sample had mp 118.5–119.5 °C; NMR δ 3.1 (t, 2 H, J = 7 Hz), 4.0 (s, 2 H), 4.1 (t, 2 H, J = 7 Hz), 7.8 (m, 4 H); IR (Nujol) 1720 cm^{-1}.

Anal. Calcd for $C_{12}H_{10}BrNO_3$: C, 48.67; H, 3.40; N, 4.73. Found: C, 49.01; H, 3.39; N, 4.56.

To a solution of 1.14 g (0.00385 mol) of **3e** and 0.535 g (0.00385 mol) of bromoacetic acid in 25 mL of Me_2CO was added 0.540 mL (0.00388 mol) of Et_3N. After 5 h a quantitative yield of Et_3N·HBr was filtered off. The residue from evaporation of the filtrate was dissolved in $CHCl_3$, and this solution was washed (1 N $NaHCO_3$ and saturated NaCl), dried ($MgSO_4$), and evaporated to give **3d** as a white solid, 1.32 g (97%).

Carbo(4-phthalimido-2-oxo)butoxymethylenetriphenylphosphonium Bromide (3f). To a solution of 5.96 g (0.0168 mol) of **3d** in benzene was added 13.41 g (0.0511 mol) of Ph_3P. After 20 h the mixture was filtered to give 10.4 g (100%) of **3f** as a bright yellow solid, mp 144–146 °C dec. An analytical sample had mp 148–148.5 °C (EtOH) and was colorless; NMR δ 2.8 (t, 2 H, J = 7 Hz), 3.9 (t, 2 H, J = 7 Hz), 4.7 (s, 2 H), 5.7 (d, J = 14 Hz), 7.8 (m, 19 H); IR (Nujol) 1720, 1740 cm^{-1}.

Anal. Calcd for $C_{32}H_{27}BrNO_5P$: C, 62.35; H, 4.41; N, 2.27. Found: C, 62.09; H, 4.82; N, 2.19.

β-(2-Phthalimidoethyl)-$\Delta^{\alpha,\beta}$-butenolide (2a). A. From β-Acetoxy-β-(2-phthalimidoethyl)-γ-butyrolactone (4). A solution of 2.59 g (0.00817 mol) of **4**[5] in 25 mL of *N,N*-diethylaniline was refluxed for 0.5 h under N_2, and the solution was poured into 100 mL of 6 N HCl with rapid stirring. The resulting suspension was cooled

and filtered, giving **2a** as an off-white solid (2.01 g, 96%), mp 180–183 °C, after drying in vacuo. An analytical sample had mp 182.5–184 °C (EtOH); NMR δ 2.8 (t, 2 H, J = 7 Hz), 3.9 (t, 2 H, J = 7 Hz), 4.8 (d, 2 H, J = 2 Hz), 5.9 (t, 1 H, J = 2 Hz), 7.8 (m, 4 H); IR (Nujol) 1760 cm^{-1}.

Anal. Calcd for $C_{14}H_{11}NO_4$: C, 65.37; H, 4.31; N, 5.45. Found: C, 65.35; H, 4.21; N, 5.47.

B. From Phosphonium Salt 3f. To a solution of 1.10 g (0.00179 mol) of **3f** in 25 mL of CH_2Cl_2 was added 0.30 mL (1.2 equiv) of freshly distilled Et_3N. After 20 h the solution was evaporated and dried in vacuo to give 1.32 g (100%) of a tan solid whose IR and NMR spectra were the sum of those of **2a**, Ph_3PO, and Et_3N·HBr. The hydrobromide was removed by treatment with H_2O, and the residue was recrystallized once from EtOH to give the product as a white solid (0.327 g, 71%), mp 176–179 °C.

Lactones 2b and 2c. The preparations of **2b** and **2c** from bromo ketones **6a** and **6b**, respectively, were analogous to that for the conversion of **3e** to **2a**, and the yields were essentially quantitative. For **2b**: mp 91–92.5 °C (lit.[16] mp 94 °C); NMR δ 5.2 (d, 2 H, J = 2 Hz), 6.3 (t, 1 H, J = 2 Hz), 7.5 (m, 5 H); IR (Nujol) 1745 cm^{-1}. For **2c**: mp 151–152.5 °C (lit.[17] mp 152–153 °C); NMR δ 6.3 (d, 1 H, J = 2 Hz), 6.6 (d, 1 H, J = 2 Hz), 7.4 (m, 10 H); IR (Nujol) 1740 cm^{-1}.

Isolation of Phosphorane 3g. A suspension of 1.00 g (0.00162 mol) of **3f** in 25 mL of Et_3N was stirred for 17 h. Evaporation of the excess amine in vacuo, stirring with H_2O, and filtration left 0.819 g (94%) of **3g** as a white solid that turned brown on standing in air. On moderately fast heating it partially melted at 97.5–99 °C, resolidified, and slowly decomposed above 120 °C; IR (Nujol) 1725, 1625 cm^{-1}.

IR Analysis of Phosphonium Salt Cyclizations. To a solution of the salt in $CHCl_3$ was added 1.2 equiv of Et_3N. The sample was immediately placed in the spectrophotometer, and the increase in adsorption of the solution at 1175 cm^{-1} (P=O) was continuously recorded via time drive to give a smooth curve; the reaction was taken to be over when the curve leveled off. The equilibrium temperature attained in the IR beam was found to be ca. 41 °C (thermocouple), but since the samples were initially at room temperature the reactions are probably somewhat faster than shown in Table I.

2-Diethylphosphonoacetoxycyclohexanone (9c). A solution of 2.38 g (0.0134 mol) of 2-bromocyclohexanone[19] and 4.12 g (0.0176 mol) of potassium diethylphosphonoacetate[20] in 50 mL of CH_2Cl_2 was allowed to stand for 4 days. Precipitated KBr was filtered off, the filtrate was evaporated, and the residue was partitioned between H_2O and $CHCl_3$. The organic layer was dried ($MgSO_4$), evaporated, and dried in vacuo to give **9c** as a pale yellow spectrally clean liquid: 3.56 g (91%); NMR δ 1.2–2.7 (m, 8 H), 1.35 (t, 6 H, J = 7 Hz), 3.09 (fine split d, 2 H, J = 1 and 21 Hz), 4.20 (fine split quintet, 4 H, J = 1 and 7 Hz), 5.19 (dd, 1 H, J = 6 and 11 Hz); IR (neat) 1750, 1730, 1265, 1025 cm^{-1}.

Registry No.—**3c**, 7504-49-6; **3d**, 66792-68-5; **3e**, 51132-00-4; **3g**, 66792-69-6; **4**, 65465-68-1; **6a**, 70-11-1; **6b**, 1484-50-0; **7a**, 53392-50-0; **7b**, 66792-70-9; **9c**, 66792-71-0; **9** (Y = Br), 822-85-5; bromoacetic acid, 79-08-3; potassium diethylphosphonoacetate, 34170-84-8.

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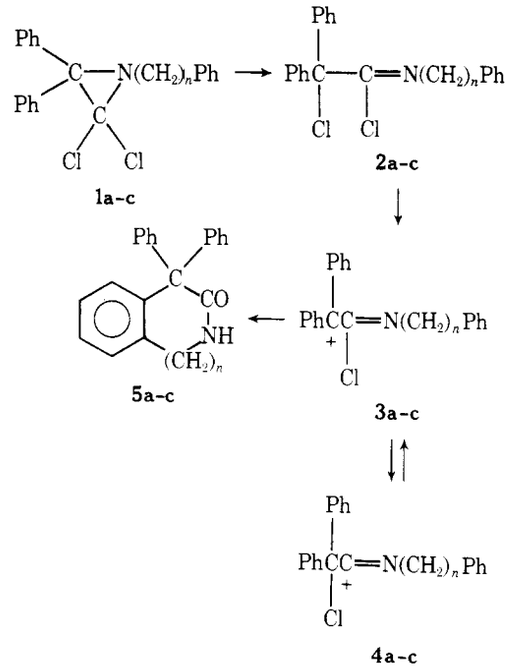
Formation of 2,3-Dihydro-2,2-diphenylbenzo[*b*]furan-3-one by Reaction of *gem*-Dichloroaziridine with Phenol: Multiplicity of Reactions of *gem*-Dichloroaziridines under Acidic Conditions

Manabu Senō,* Shinsaku Shiraishi, Hideo Kise, and Yukio Suzuki

Institute of Industrial Science, University of Tokyo, Roppongi 7-22, Minato-ku, Tokyo 106, Japan

Received March 21, 1978

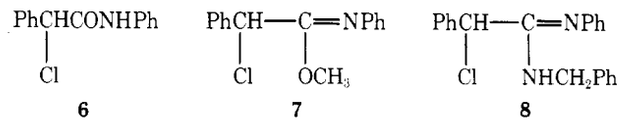
We have observed the formation of 4,4-diphenyl-1,2,3,4-tetrahydroisoquinolin-3-one (**5b**) and 1,1-diphenyl-1,3,4,5-tetrahydro-2*H*-3-benzazepin-2-one (**5c**) by treatment of 1-



a, n = 0; **b**, n = 1; **c**, n = 2

benzyl-2,2-dichloro-3,3-diphenylaziridine (**1b**) and 2,2-dichloro-3,3-diphenyl-1-(2-phenylethyl)aziridine (**1c**), respectively, with sulfuric acid in acetic acid.[1] It was assumed that the reactions proceed through cleavage of the C(3)–N bond and subsequent or simultaneous migration of a chlorine atom to the C(3) position to form *N*-benzyl- or *N*-(2-phenylethyl)-α-chloro-α,α-diphenylacetimidoyl chloride (**2b** or **2c**). The subsequent intramolecular Friedel–Crafts reaction and hydrolysis give the cyclic compounds **5b** or **5c**. In the case of *N*-phenyl compound **1a**, the intermediate (**2a**) was isolated by thermal isomerization. It was converted to 3,3-diphenyloxindole (**5a**) in good yield by treatment with sulfuric acid–acetic acid.[1,2]

Since the hydrolysis, methanolysis, and aminolysis of 2,2-dichloro-1,3-diphenylaziridine were reported to give compounds **6**,[3] **7**,[4] and **8**,[5] both of the two chlorine-carrying



carbon atoms in **2** are considered to be sensitive to nucleophilic attack. Therefore, we expected the formation of heterocyclic compounds by intermolecular reactions of **1** with nucleophilic aryl compounds. We wish now to report the formation of 2,3-dihydro-2,2-diphenylbenzo[*b*]furan-3-one (**12**) by the reaction of **1c** with phenol.

When **1c** was reacted with phenol in benzene in the presence

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