Table I. ^{1}H NMR Spectral Data in Me_2SO-d_6

	chemical shift, δ								coupling constant, Hz				
compd	H-1	H-2	H-3	H-5	H-6	H-7	H-9	H-10	$J_{1,2}$	$J_{2,3}$	$J_{5,6}$	$J_{7,9}$	$J_{9,10}$
2				7.94	8.40	8.49	8.12	8.94			9.5	2.0	8.6
3		8.20	8.84		8.3–8.5		7.94	9.15		1.3		2.0	8.6
4	7.70	8.56			8.4–8.5		8.04	9.22	2.6			2.0	8.6
5		9.12			8.6–8.7		8.18	9.38				2.2	8.6

protons (the two high-field doublets and the two downfield doublets, respectively). There is no evidence for **2a**.

A last point to consider is whether the bromo substituent plays an important role in the stability of **2c**. As the 200-MHz ^{1}H NMR spectrum of **1** in Me_2SO-d_6 at 20 °C does agree with that of **2** under the same conditions, i.e., since the only tautomer detected is **1c**, it may be thought that its role is not significant.

In conclusion we can remark, correcting the previous suggestions on the subject, that the tetrazolo[1,5-*b*]-*as*-triazine system (as in **1c** and **2c**) is largely favored with respect to the tetrazolo[5,1-*c*]-*as*-triazine arrangement (as in **1a** and **2a**) in the naphtho series considered here. Indeed, this agrees with what is observed in simple *as*-triazine systems, in which the compounds cyclized on N-2 (tetrazolo[1,5-*b*]-*as*-triazines) usually predominate over those cyclized on N-4 (tetrazolo[5,1-*c*]-as-triazines),[2,7] but partially disagrees with what occurs in the benzo-*as*-triazine series.[3] A general explanation for all these facts could be as follows: the cyclization of the azide on N-2 seems to be favored as a rule,[7] probably because the lone-pair repulsion between N-1 and N-2 disappears after such a cyclization, but may give more quinone-like or less aromatic polycyclic structures than the cyclization on N-4; the relative importance of this last factor may in some cases, particularly in the benzo series, invert the stability order.

Experimental Section

Melting points are uncorrected. ^{1}H NMR spectra were recorded on Varian XL-200 (200 MHz) and Perkin-Elmer R-24 or R-12B spectrometers; chemical shifts are reported in parts per million with respect to internal Me_4Si in all the cases, and *J* values are given in hertz. IR spectra were obtained on a Perkin-Elmer 283 instrument.

Azo derivatives [(6-bromo-2-hydroxynaphthalen-1-yl)-1-azoazoles] were prepared by standard procedures,[1] from 6-bromo-2-naphthol and the diazo derivatives arising from 5-aminotetrazole hydrate, 2-aminoimidazolium sulfate, 3(5)-aminopyrazole, and 3(5)-amino-*s*-triazole and were used in the next step without further purification. Dehydration of 2-(2-hydroxynaphthalen-1-yl)-1-azo-5-tetrazole to naphtho[2,1-*e*]tetrazolo[5,1-*c*]-*as*-triazine and/or related tautomers (**1**) was carried out as reported.[1]

8-Bromonaphtho[2,1-*e*]tetrazolo[1,5-*b*]-*as*-triazine (2c). 2-(6-Bromo-2-hydroxy-naphthalen-1-yl)-1-azo-5-tetrazole (5.0 g, 15.7 mmol) was heated at reflux in 2 M aqueous H_2SO_4 for 2 h. The resulting solution was basified with 2 M aqueous NaOH and then continuously extracted with CH_2Cl_2. The organic extracts were dried over anhydrous Na_2SO_4, filtered, and evaporated under vacuum to afford 3.8 g (80% yield) of almost pure **2**, which was recrystallized in ethanol to give a yellow amorphous solid: mp 202–204 °C; IR (KBr) 3060, 1585, 1525, 1425, 1345, 1295, 1275, 1125, 1110, 1030, 970, 885, 835 cm^{-1}; IR ($CHCl_3$) 3030, 2130, 2110, 1595, 1520, 1465, 1400, 1365, 1300, 1230, 1070 cm^{-1}; 200 MHz ^{1}H NMR ($CDCl_3$) δ 7.75 (d, *J* = 9.2 Hz, H-5), 7.96 (dd, *J* = 2.2 Hz, *J* = 8.6 Hz, H-9), 8.07 (d, *J* = 9.2 Hz, H-6), 8.09 (d, *J* = 2.2 Hz, H-7), and 9.24 (d, *J* = 8.6 Hz, H-10), corresponding to **2b**, and δ 7.86 (d, *J* = 9.6 Hz, H-5), 8.01 (dd, *J* = 2.2 Hz, *J* = 8.6 Hz, H-9), 8.11 (d, *J* = 2.2 Hz, H-7), 8.13 (d, *J* = 9.6 Hz, H-6), and 9.00 (d, *J* = 8.6 Hz, H-10), corresponding to **2c**. Anal. Calcd for $C_{11}H_7N_6OBr$: C, 41.40; H, 2.21; N, 26.34. Found: C, 41.17; H, 2.48; N, 26.01.

8-Bromonaphtho[2,1-*e*]azolo-*as*-triazines 3–5. Solutions of 4–5 g of crude 2-(8-bromo-2-hydroxynaphthalen-1-yl)-1-azoazoles in ca. 100 mL of acetic acid were refluxed for 30 min (imidazole derivative), 24 h (pyrazole derivative), and 48 h (*s*-triazole derivative). Elimination of the solvent under vacuum and recrystallization of the residues from absolute EtOH gave **3–5**, respectively.

8-Bromonaphtho[2,1-*e*]imidazo[2,1-*c*]-*as*-triazine (**3**): 58% yield (from the starting amine); mp >300 °C; IR (KBr) 3140, 3060, 1585, 1520, 1465, 1440, 1395, 1340, 1300, 1170, 1120, 1100, 1060, 890, 870, 820, 780, 730 cm^{-1}.

8-Bromonaphtho[2,1-*e*]pyrazolo[5,1-*c*]-*as*-triazine (**4**): 85% overall yield; mp 234–236 °C; IR(KBr) 3060, 2920, 1585, 1530, 1510, 1465, 1420, 1400, 1335, 1320, 1275, 1140, 1065, 895, 830, 820, 750, 720 cm^{-1}.

8-Bromonaphtho[2,1-*e*]-*s*-triazolo[5,1-*c*]-*as*-triazine (**5**): 87% overall yield; mp 287–288 °C; IR (KBr) 3060, 1585, 1530, 1460, 1400, 1315, 1290, 1240, 1200, 1170, 1130, 910, 895, 825 cm^{-1}.

Registry No. 2b, 81940-10-5; **2c**, 81940-11-6; **3**, 81940-12-7; **4**, 81940-13-8; **5**, 81940-14-9; 6-bromo-2-hydroxynaphthalene-1-azo-5′-tetrazole, 81940-15-0.

(7) (a) Weissberger, A.; Taylor, E. C. *Chem. Heterocycl. Compd.* **1977**, *30*, 179. (b) Goodman, M. M.; Atwood, J. L.; Carlin, R.; Hunter, W.; Paudler, W. W. *J. Org. Chem.* **1976**, *41*, 2860. (c) Goodman, M. M.; Paudler, W. W. *Ibid.* **1977**, *42*, 1866.

Active Heteromethylene Compounds. 2.[1a,b] A New Synthesis of *N*-(Halomethyl)acylamides

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Received March 9, 1982

Although a number of reports relate to the preparation of *N*-(halomethyl)acylamides, including some quite recent references,[2] known methods still possess severe limitations in gaining access to these desirable intermediates. Thus, *N*-(chloromethylation) with combinations of formaldehyde and HCl[3] is narrowly confined to simple aliphatic amides, and even here mixtures and disappointing yields are most often encountered. Although the conversion of *N*-(hydroxymethyl) amides with phosphorus pentachloride, thionyl chloride, or halogen acids is convenient, this method is largely limited to substrates derived from reaction of formaldehyde with imides or primary amides, owing to incomplete reaction and side products when secondary amides or anilides are methylolated.[4] Similarly, the addition of acid chlorides to monomeric[5,6] or trimeric[2a,7]

(1) (a) Part I: K. W. Ratts and J. P. Chupp, *J. Org. Chem.*, **39**, 3745 (1974); (b) presented at the 183rd National Meeting of the American Chemical Society, Las Vegas, NV, April 1, 1982; ORGN 195.

(2) (a) S. Gronowitz and Z. Lidert, *Syntheses*, 810 (1979); (b) J. W. Worley, *J. Org. Chem.*, **44**, 1178 (1979); and references contained in both these citations.

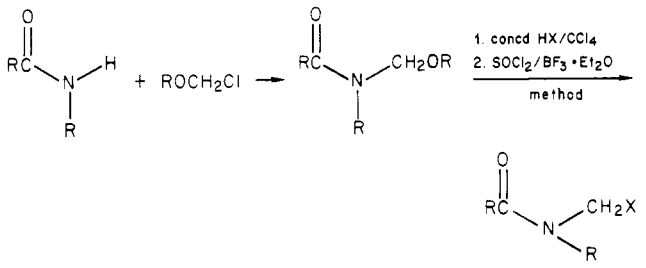
(3) A. W. Baldwin and H. A. Piggott, U.S. Patent 2 131 362 to Imperial Chemical Industries, Ltd., 1938.

(4) J. P. Chupp and A. J. Speziale, *J. Org. Chem.*, **28**, 2592 (1963).

(5) J. P. Chupp, J. F. Olin, and H. K. Landwehr, *J. Org. Chem.*, **34**, 1192 (1969).

0022-3263/82/1947-3169$01.25/0

Scheme I[a]



[a] See examples in the Experimental Section for definition of R, X, and method (1 or 2).

azomethines can only be conveniently applied to electron-rich amines or anilines in which the azomethine substrate arising from formaldehyde condensation can be discretely formed in high yields. Finally, recent studies[8,9] have revealed the conversion of certain *N*-methyl amides to *N*-(chloromethyl) amides by free-radical (*hν*)-chlorination. Again, the method is confined to substrates not having other sensitive halogenation sites.

By utilizing recent advances in amide N-alkylation, particularly under phase-transfer conditions,[10-12] the facile preparation of *N*-(alkoxymethyl)acylamides from secondary acylamides, halomethyl ethers, and liquid caustic can now be easily achieved, particularly for the more acidic substrates[13] such as secondary anilides and 1-enamides. It has now been found that action of concentrated halogen acids, including 37% HCl on such *N*-(alkoxymethyl) amides easily produce the desired *N*-(halomethyl) derivatives. This is surprising since these "amidacetals" can be notoriously acid sensitive, hydrolyzing to the secondary amide, formaldehyde, and alcohol.

In favorable circumstances, merely shaking or stirring the substrate amide, dissolved in carbon tetrachloride, with concentrated HCl is enough to cause complete conversion to *N*-(chloromethyl) amide (as determined by ^{1}H NMR). In other instances it is necessary to both heat and stir the two-phase system, sometimes for prolonged periods, to cause complete reaction of amide substrate. In any case, purification is easily effected by layer separation, with subsequent removal of organic solvent. Usually, recrystallization or distillation of the resulting *N*-(halomethyl) amide residue can be carried out, although the crude product as obtained is generally quite pure enough for further syntheses.

As mentioned above, however, these *N*-(alkoxymethyl) amides are also susceptible to acid hydrolysis to secondary amide. This latter reaction becomes competitive with *N*-(halomethyl) formation in amides where facile oxygen–methylene cleavage is attenuated by the presence of electron-withdrawing groups which destabilize formation of the intermediate amidomethylene carbocation. Likewise, 1-enamides can be sensitive to acid hydrolysis, reverting to parent aldehyde or ketone.

Consequently, a further method was devised with excess thionyl chloride as reactant and solvent in the presence of a Lewis acid (BF_3·Et_2O is convenient; Scheme I) This method has the advantage of eliminating traces of moisture, as the solvent serves admirably as a water scavenger. The acid catalyst is necessary, however, apparently to activate amidomethylene–oxygen cleavage, and so facilitate chlorine transfer from the reactive solvent. Temperatures up to the reflux temperature of thionyl chloride can be chosen to conveniently effect conversion.

Experimental Section

General Procedures. Melting points were determined on a Mel-Temp apparatus or Fisher-Johns block and are uncorrected. Nuclear magnetic resonance spectra were recorded on a Varian T-60 or EM-360 spectrometer. Elemental analyses were performed by Atlantic Microlab, Inc., and Industrial Testing Laboratories (St. Louis, MO).

α-Chloro-*N*-(bromomethyl)-*N*-(2,6-dimethyl-1-cyclohexen-1-yl)acetamide. *N*-(Methoxymethyl)-*N*-(2,6-dimethyl-1-cyclohexen-1-yl)chloracetamide[12] (2 g) was dissolved in 20 mL of carbon tetrachloride and shaken with 10 mL of 48% HBr. ^{1}H NMR revealed only ca. 75% conversion even with additional mixing. After layer separation a fresh 10-mL concentrated HBr wash completed the reaction. The organic (lower) layer was separated and vacuum treated to remove solvent, traces of HBr, moisture, etc., leaving 1.7 g (75% yield) of oil which crystallized on standing to essentially pure product. Recrystallization from hexane provided the analytical sample: mp 52–53 °C; ^{1}H NMR ($CDCl_3$) δ 1.05 (d, 3 H, J = 7 Hz >$CHCH_3$), 1.7 (br s, 3 H, =CCH_3), 1.7–2.9 (3 br envelopes, 7 H, cyclohexyl H), 4.2 (s, 2 H, $ClCH_2CO$), 5.4 (AB q, 2 H, J = 7 Hz, NCH_2Br).

Anal. Calcd for $C_{11}H_{17}BrClNO$: C, 44.84; H, 5.82; N, 4.75. Found: C, 44.78; H, 5.84; N, 4.77.

α-Chloro-*N*-(chloromethyl)-*N*-(2,6-dimethyl-1-cyclohexen-1-yl)acetamide. *N*-(Ethoxymethyl)-*N*-(2,6-dimethyl-1-cyclohexen-1-yl)chloracetamide[12] (3.2 g, 0.012 mol) was dissolved in 30 mL of carbon tetrachloride and the solution stirred magnetically in an Erlenmeyer flask with 30 mL or 37% HCl solution for 1 h. ^{1}H NMR indicated complete reaction. The layers were separated, and the organic phase was washed once with cold 10% HCl, dried over $MgSO_4$, and evaporated in vacuo [75 °C (0.2 mm)] to give 2.5 g of clear oil (81% yield). One gram of this material was subjected to bulb-to-bulb distillation at 110 °C (0.02 mm) to give 0.85 g of clear oil which completely solidified. The distillation residue weighed 0.15 g. Recrystallization of the distillate from hexane afforded colorless crystals: mp 40–40.5 °C; ^{1}H NMR ($CDCl_3$) δ 1.0 (d, 3 H, J = 7 Hz, >$CHCH_3$), 1.62 (br s, 3 H, =CCH_3), 1.4–2.8 (3 br envelopes, 7 H, cyclohexyl H)8 4.05 (s, 2 H, $ClCH_2CO$), 5.25 (AB q, 2 H, J = 7 Hz, NCH_2Cl).

Anal. Calcd for $C_{11}H_{17}Cl_2NO$: C, 52.81; H, 6.85; Cl, 28.34; N, 5.60. Found: C, 52.81; H, 6.89; Cl, 28.30; N, 5.57.

α-Chloro-2′-ethyl-6′-(trifluoromethyl)-*N*-(chloromethyl)acetanilide. α-Chloro-2-ethyl-6-(trifluoromethyl)-*N*-(methoxymethyl)acetanilide (14.8 g) was dissolved in 100 mL of thionyl chloride and ca. 0.2 mL of boron trifluoride etherate was added thereto. The temperature was raised to reflux. It was necessary to heat this mixture at that temperature for ca. 24 h to effect complete reaction as determined by ^{1}H NMR. The thionyl chloride was removed under vacuum, and methylene chloride was added to the residue and vacuum treated once again. Finally, in methylene chloride once again the material was washed with 37% HCl, dried over $MgSO_4$, and filtered, and the solvent was removed under vacuum. The residue was recrystallized from cold hexane–ether to give 11.7 g (78% yield) of crystals: mp 46–50 °C; ^{1}H NMR ($CDCl_3$) δ 1.4 (t, 3 H, J = 7 Hz, CH_2CH_3), 2.90 (m, (AB q, t) 2 H, J = 7 Hz, CH_2CH_3), 3.80 (s, 2 H, $ClCH_2CO$), 5.5 (AB q, 2 H, J = 8 Hz, NCH_2Cl), 7.75 (m, 3 H, Ar H).

Anal. Calcd for $C_{12}H_{12}Cl_2F_3NO$: C, 45.88; H, 3.85; N, 4.46. Found: C, 45.89; H, 3.89; N, 4.45.

α-Chloro-2′-isobutoxy-*N*-(chloromethyl)acetanilide. α-Chloro-2′-isobutoxy-*N*-(methoxymethyl)acetanilide (5.9 g) was dissolved in 100 mL of thionyl chloride to which 4 drops of boron trifluoride etherate had been added and the mixture was refluxed for 1.5 h. The thionyl chloride was removed under vacuum, toluene was added, and the solution was vacuum treated again to remove solvent. The residue was then taken up in ether, washed

(6) H. Böhme and K. Hartke, *Chem. Ber.*, **96**, 600 (1963).
(7) Canadian Patent 779917 to Monsanto Co., 1968.
(8) H. Ulrich, R. Richter, P. J. Whitman, and A.A.R. Sayigh, *J. Org. Chem.*, **39**, 2897 (1974).
(9) H. Böhme, J. P. Denis, and H. J. Drechsler, *Ann. Chem.*, 1447 (1979).
(10) R. Brehme, *Synthesis*, 113–14 (1976).
(11) U. Schöllkopf and H. Beckhaus, *Angew. Chem., Int. Ed. Engl.* **15**, 293 (1976).
(12) U.S. Patent 4258196 to Monsanto Co., 1981.
(13) The less acidic saturated aliphatic or cycloaliphatic amides can be similarly treated, but resort to more basic amide alkylation conditions (i.e., Na, NaH, K, see ref 12) is often required.

with 10% HCl, and then dried over $MgSO_4$. After filtration and vacuum removal of solvent, the residual oil was distilled in a bulb-to-bulb apparatus at 137 °C (0.15 mm) to give 5.0 g (86% yield) of a yellow oil: ^1H NMR ($CDCl_3$) δ 0.93 (d, 6 H, J = 7 Hz, $CH(CH_3)_2$), 2.0 (heptet, 1 H, J = 7 Hz, $CH(CH_3)$), 3.72 (d, 2 H, J = 7 Hz, CH_2CH), 3.8 (s, 2 H, $ClCH_2CO$)8 5.40 (AB q, 2 H, J = 8 Hz, NCH_2Cl), 6.7–7.6 (envelopes, 4 H, Ar H)

Anal. Calcd for $C_{13}H_{17}Cl_2NO_2$: C, 53.81; H, 5.91; N, 4.83. Cl, 24.43. Found: C, 53.85; H, 5.95; N, 4.83; Cl, 24.34.

Registry No. α-Chloro-*N*-(bromomethyl)-*N*-(2,6-dimethyl-1-cyclohexen-1-yl)acetamide, 81634-03-9; *N*-(methoxymethyl)-*N*-(2,6-dimethyl-1-cyclohexen-1-yl)chloracetamide, 78179-95-0; α-chloromethyl-*N*-(2,6-dimethyl-1-cyclohexen-1-yl)acetamide, 81634-02-8; *N*-(ethoxymethyl)-*N*-(2,6-dimethyl-1-cyclohexen-1-yl)chloroacetamide, 77117-40-9; α-chloro-2′-ethyl-6′-(trifluoromethyl)-*N*-(chloromethyl)acetanilide, 81634-12-0; α-chloro-2′-ethyl-6′-(trifluoromethyl)-*N*-(methoxymethyl)acetanilide, 80808-80-6; α-chloro-2′-isobutoxy-*N*-(chloromethyl)acetanilide, 81987-75-9; α-chloro-2′-isobutoxy-*N*-(methoxymethyl)acetanilide, 81987-76-0.

Synthesis of Catalpalactone

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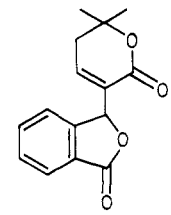
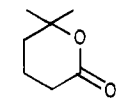
Received February 4, 1982

Catalpalactone, a dilactone of natural occurrence, is found in the heartwood of the tree *Catalpa ovata* G. Don, from which it was isolated by Nagakura and co-workers.[1] Later the same compound was encountered in the wood of a related species, *C. bignonioides* Walt.[2] The structure 1 for catalpalactone has been deduced on the basis of chemical behavior and spectroscopic properties.[1,2] Despite the presence of an asymmetric center in 1, the natural compound is optically inactive and presumably a racemate.

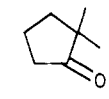
We envisaged a synthesis of catalpalactone via alkylation of δ,δ-dimethyl-δ-valerolactone (2) by a 3-substituted phthalide in which the substituent is a good leaving group. The lactone was obtained by reduction of commercial 5,5-dimethyl-2-pentenolactone, by Baeyer–Villiger oxidation of 2,2-dimethylcyclopentanone (3),[3] and from 2-methyl-3-buten-2-ol by ethyl cyanoacetate addition followed by hydrolysis and lactonization.[4] The Baeyer–Villiger reaction was expected to furnish the desired lactone on mechanistic grounds.[5] We first chose 3-bromophthalide (4), readily available by free-radical bromination of phthalide,[6] as alkylating agent. Exposure of it to the anion of δ,δ-dimethyl-δ-valerolactone in dry tetrahydrofuran afforded dihydrocatalpalactone (5) in 8% yield, the product having melting point and spectral properties identical with those reported;[1,2] an authentic sample was not available. Apparently this reaction was stereoselective, only one of the two possible racemates having been formed. No attempt was made to establish its stereochemistry, since the next step destroyed one of the centers of asymmetry. A greatly superior yield (78%) of 5 was obtained when 3-[(*p*-toluenesulfonyl)oxy]phthalide (6) was used as substrate.



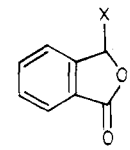
1

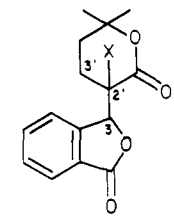


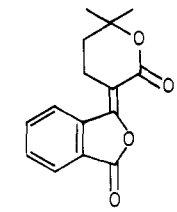
2



3







4, X = Br
6, X = $OSO_2C_6H_4CH_3(p)$

5, X = H
7, X = SePh

8

The dihydrocatalpalactone was next converted into its carbanion with lithium 2,2,6,6-tetramethylpiperidide. Because of the outcome of the synthetic sequence (see below) the carbanion center must be located at position 2′ rather than 3 in 5. The carbanion was reacted with diphenyl diselenide[7] to give, in 15% yield, 2′-(phenylseleno)dihydrocatalpalactone (7), along with considerable amounts of unreacted diselenide and lactone, presumably because of steric hindrance of the reaction. This product was subjected to elimination vis its selenoxide, by treatment with hydrogen peroxide. This gave, in quantitative yield from the selenide, catalpalactone (1), identical in every respect with an authentic sample. None of the isomeric product 8 was encountered, elimination having occurred exclusively via the 3′- rather than the 3-position in 7. This behavior was to be expected because elimination away from an electronegative atom (in this case oxygen) is generally preferred; further, the formation of an endocyclic rather an exocyclic double bond is favored.[8]

Experimental Section

Melting points and boiling points are uncorrected. UV and IR spectra were recorded on Perkin-Elmer 202 and 137 instruments, respectively. NMR spectra were measured on a JEOL FX-90Q instrument and mass spectra on a Hewlett-Packard 5840A GC/MS spectrometer. GC was conducted on an F and M Model 810 gas chromatograph.

3-[(*p*-Toluenesulfonyl)oxy]phthalide (6). 3-Bromophthalide was prepared by the free-radical bromination of phthalide,[6] in 73% yield. It crystallized from cyclohexane in plates: mp 74–78 °C (lit.[6] mp 78–80 °C); NMR ($CDCl_3$) δ 7.1 (s, 1 H, CHBr), 7.3–7.8 (m, 4 H, Ar H). This compound (2.13 g, 0.01 mol) and freshly prepared, dry silver *p*-toluenesulfonate[9] (2.79 g, 0.01 mol) in dry acetonitrile (50 mL) were stirred at 0 °C for 1 h and then filtered with the aid of Celite. The solvent was removed in vacuo, leaving a colorless syrup (3.05 g, 100%) which solidified readily and showed a single spot on TLC. It was used without further purification. 6: IR ($CHCl_3$) 1750 (C=O), 1390 (CSO_2OR) cm^{-1}; NMR ($CDCl_3$ + Me_2SO-d_6) δ 2.2 (s, 3 H, CH_3), 7.1 (s, 1 H, CHO), 7.0–7.8 (m, 4 H, Ar H). Attempts to make this compound by conventional tosylation of 3-hydroxyphthalide were unsuccessful owing to formation of di-3-phthalyl ether by tosylate displacement by the anion of the substrate.

δ,δ-Dimethyl-δ-valerolactone (2). A. Commercial 5,5-dimethyl-2-pentenolactone (0.89 g, 0.007 mol) in ethanol (75 mL) was shaken with 10% Pd–C catalyst (0.1 g) in hydrogen at at-

(1) Inouye, H.; Okuda, T.; Hirata, Y.; Nagakura, N.; Yoshizaki, M. *Tetrahedron Lett.* **1965**, 1261.
(2) Inouye, H.; Okuda, T.; Hirata, Y.; Nagakura, N.; Yoshizaki, M. *Chem. Pharm. Bull. Jpn.* **1967**, *15*, 786.
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(4) Surzur, J. M.; Teissier, P. *Bull. Soc. Chim. Fr.* **1970**, 653, 1611.
(5) Norman, R. O. C. "Principles of Organic Synthesis", 2nd ed.; Chapman and Hall: London, 1978; p 471.
(6) Koten, I. A.; Sauer, R. J. "Organic Syntheses", Collect. Vol. V; Wiley: New York, 1973; p 145.
(7) Cf. Grieco, P. A.; Miyashita, M. *J. Org. Chem.* **1974**, *39*, 120.
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