$C_8H_{10}O_2$: M_r 138.0681. Found: M_r (mass spectrum) 138.0684.

β-Methylene-α,α-tetramethylene-γ-butyrolactone (3e):[2d,e] ^1H NMR ($CDCl_3$) δ 1.6–2.4 (m, 8 H), 4.80 (t, J = 2 Hz, 2 H), 5.06 (m, 2 H); IR (film) 3075, 1770, 1670, 1020, 900 cm^{-1}; mass spectrum, m/e 152 (M^+).

β-Methylene-α,α-pentamethylene-γ-butyrolactone (3f):[2b,f,g] mp 41–42 °C (lit. mp 46 °C,[2b] 42–43 °C[2g]); ^1H NMR ($CDCl_3$) δ 1.3–2.2 (m, 10 H), 4.78 (t, J = 2 Hz, 2 H), 5.09 (t, J = 2 Hz, 1 H), 5.18 (t, J = 2 Hz, 1 H); IR (film) 3070, 1770, 1665, 1020, 900 cm^{-1}; mass spectrum, m/e 166 (M^+).

Acknowledgment. We thank Professor A. Rassat and Dr. J. L. Luche for their interest in this work, the CNRS (LA 332) for financial support, and the C.N.Pq. for a fellowship award to F.C.

Registry No. 1, 95864-49-6; 2 (R = CH_3, R′ = H), 95864-50-9; 2 (R = $(CH_3)_2CH$, R′ = H), 95864-51-0; 2 (R = CH_2=$CHCH_2$), 95864-52-1; **2a**, 95892-00-5; **2c**, 95864-56-5; **2d**, 95864-53-2; **2e**, 95864-54-3; **2f**, 95864-55-4; **3a**, 73461-19-5; **3b**, 95864-57-6; **3c**, 95864-58-7; **3d**, 95864-59-8; **3e**, 57429-72-8; **3f**, 63965-86-6; CH_3I, 74-88-4; $(CH_3)_2CHI$, 75-30-9; CH_2=$CHCH_2Br$, 106-95-6; $Br(CH_2)_3Br$, 109-64-8; $Br(CH_2)_4Br$, 110-52-1; $Br(CH_2)_5Br$, 111-24-0; CH_3OCH_2Cl, 107-30-2; diazomethane, 334-88-3; *tert*-butyldimethylsilyl chloride, 18162-48-6; (*E*)-4-hydroxy-3-methyl-2-butenoic acid, 44647-19-0; methyl (*E*)-4-hydroxy-3-methyl-2-butenoate, 13866-57-4; 2-isopropyl-3-methylbut-2-enolide, 95864-60-1; 2 (R = $Br(CH_2)_3$, R′ = H), 96020-93-8; 2 (R = $Br(CH_2)_4$, R′ = H), 96020-94-9; 2 (R = $Br(CH_2)_5$, R^1 = H), 96020-95-0.

A Direct Synthesis of [(*tert*-Butoxycarbonyl)methylidene]azacycloalkanes from *N*-Alkyl Lactams

Masahiko Yamaguchi* and Ichiro Hirao

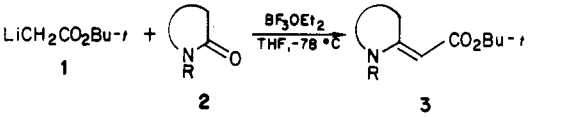
Department of Industrial Chemistry, Kyushu Institute of Technology, Sensui-cho, Tobata, Kitakyushu 804, Japan

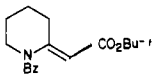
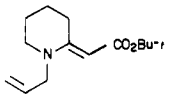
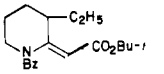
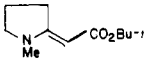
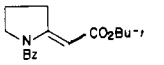
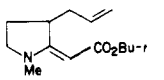
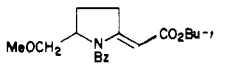
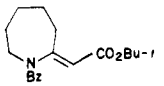
Received September 11, 1984

[(Alkoxycarbonyl)methylidene]azacycloalkanes **3** are useful compounds for the synthesis of various nitrogen-containing natural products. These compounds have been prepared by Knoevenagel reactions on lactam-derived acetals,[1] imino ethers,[2] iminium chlorides,[3] or (alkylthio)alkylidenium salts[4] followed by decarboxylation. Other routes include Eschenmoser's sulfide-contraction procedure via thiolactams[5] and a novel Wittig reaction of *N*-sulfonyl lactams.[6] These procedures, however, require the conversion of lactams to activated derivatives, and overall yields are often low.[7]

During our studies on new synthetic methods utilizing the combination of organolithium compounds and BF_3·OEt_2,[8] we found that lithium *tert*-butyl accetates **1** readily reacts with *N*-alkyl lactams **2** in the presence of this Lewis acid to give the enamino esters **3** (Table I). The details of this investigation are described herein.

Table I.
2-[(*tert*-Butoxycarbonyl)methylidene]azacycloalkanes 3



3		yield,[a] %
3a		54 (quant)[b]
3b		42
3c		30
3d		48, 47[c]
3e		45 (87)[b]
3f		21
3g		28
3h		41[d]

[a] Isolated yields are given. [b] Conversion based on starting material consumed. [c] The reaction was carried out with 2.5 mmol of **2d** and 5 mmol of *tert*-butyl acetate. [d] The reaction was quenched with piperidine and the product was isolated by chromatography on alumina.

The lithium salt **1** was derived from *tert*-butyl acetate and lithium diisopropylamide (LDA) in THF at −78 °C. Treatment of **1** with 1-benzyl-2-piperidone (**2a**) followed by BF_3·OEt_2 for 30 min at −78 °C, gave 1-benzyl-2-[(*tert*-butoxycarbonyl)methylidene]piperidine (**3a**) in 54% yield. Unchanged starting material could be recovered, and the conversion to **3a** based on consumed **2a** was high. The reaction of **1** with BF_3·OEt_2 seems to be competitive as the addition of BF_3·OEt_2 prior to **2a** did not give **3a** at all. Stereochemical assignments are based on ^1H NMR shift reagent studies, using tris(dipivalomethanato)europium(III) ($Eu(DPM)_3$). With $Eu(DPM)_3$ large deshielding effects were observed for C-3 hydrogens of **3a**, indicating it to be the *E* isomer.[2c,9]

Various reaction conditions were investigated in the synthesis of 1-allyl-2-[(*tert*-butoxycarbonyl)-methylidene]piperidine (**3b**). The use of THF as the solvent is essential; other solvents such as ether, toluene, or hexane gave no detectable amount of **3b**. The use of $TiCl_4$ instead of BF_3·OEt_2 as the Lewis acid gave, as well as enamine **3b** (33%), 1-allyl-2,2-bis[(*tert*-butoxycarbonyl)methyl]piperidine (**4b**) in 35% yield. The dialkylated piperidine **4b** is considered to be formed by attack of **1** on the iminium intermediate. However, we

(1) (a) Meerwein, H.; Florian, W.; Schön, N.; Stopp, G. *Justus Liebigs Ann. Chem.* **1961**, *641*, 1. (b) Shov, Y.; Shanan-Atidi, H. *J. Am. Chem. Soc.* **1969**, *91*, 6683, 6689.

(2) (a) Horii, Z.; Morikawa, K.; Ninomiya, I. *Chem. Pharm. Bull.* **1969**, *17*, 2230. (b) Oishi, T.; Nagai, M.; Onuma, T.; Moriyama, H.; Tsutae, K.; Ochiaia, M.; Ban, Y. *Ibid.* **1969**, 17. 2306. (c) Célérier, J.-P.; Deloisy, E.; Lhommet, G.; Maitte, P. *J. Org. Chem.* **1979**, *44*, 3089.

(3) Bredereck, H.; Bredereck, K. *Chem. Ber.* **1961**, *94*, 2278.

(4) (a) Restle, S.; Wermuth, C. G. *Tetrahedron Lett.* **1979**, 4837. (b) Gugelchuk, M. M.; Hart, D. J.; Tsai, Y.-M. *J. Org. Chem.* **1981**, *46*, 3671.

(5) Roth, M.; Dubs, P.; Götschi, E.; Eschenmoser, A. *Helv. Chim. Acta.* **1971**, *54*. 710.

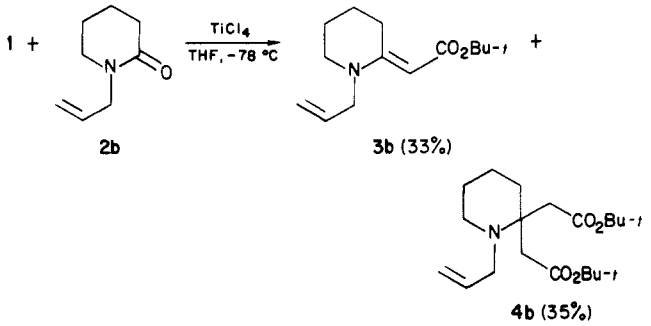
(6) Natsume, M.; Takahashi, M.; Kiuchi, K.; Sugaya, H. *Chem. Pharm. Bull.* **1971**, *19*, 2648.

(7) A reaction of lithio-*N*,*N*-dimethyl(trimethylsily)acetamide with a lactam is known: Woodbury, R. P.; Rathke, M. W. *Tetrahedron Lett.* **1978**, 709.

(8) (a) Yamaguchi, M.; Hirao, I. *Tetrahedron Lett.* **1983**, *24*, 391, 1719. (b) Yamaguchi, M.; Waseda, T.; Hirao, I. *Chem. Lett.* **1983**, 35. (c) Yamaguchi, M.; Nobayashi, Y.; Hirao, I. *Tetrahedron Lett.* **1983**, *24*, 5121. (d) Yamaguchi, M.; Shibato, K.; Hirao, I. *Ibid.* **1984**, *24*, 1159. (e) Yamaguchi, M.; Hirao, I. *J. Chem. Soc., Chem. Commun.* **1984**, 202.

(9) Krueger, S. A.; Bryson, T. A. *J. Org. Chem.* **1974**, 39, 3167.

0022-3263/85/1950-1975$01.50/0



were not able to synthesize **4b** selectively by varying solvents, temperature, or molar ratio of reagents.

In the reaction of *N*-alkylpyrrolidines with **1** under the same conditions, it was necessary to treat the reaction mixture with an amine prior to aqueous workup in order to obtain the products in reasonable yields. Pyrrolidine was the best of amines tested, and more strongly coordination amines such as tetramethylethylenediamine, *N*-methylpiperidine, or morpholine gave lower yields (Table II). However, the yields of piperidine derivatives **3** were not increased by treatment of the reaction mixtures with pyrrolidine.

1-Benzyl-2-[(*tert*-butoxycarbonyl)methylidene]perhydroazepine (**3h**) was isolated by chromatography on alumina because it decomposed rapidly on silica gel to give the starting lactam **2h**.

Thus the reaction provides the first route to enamines **3** directly from lactams.

Reaction of acyclic amide *N,N*-dimethylhexanamide (**5**) with **1** under the same reaction conditions gave *tert*-butyl 3-oxooctanoate (**6**) in 30% yield with loss of the amino group; no enamino ester was detected.

$$1 + \underset{\mathbf{5}}{n\text{-}C_5H_{11}CONMe_2} \xrightarrow[\text{THF, -78 °C}]{BF_3\cdot OEt_2} \underset{\mathbf{6}\ (30\%)}{n\text{-}C_5H_{11}COCH_2CO_2Bu\text{-}t}$$

Experimental Section

Melting points are uncorrected. ^{1}H NMR spectra were determined on a JEOL JNM-FX-60 spectrometer in $CDCl_3$, with Me_4Si as the internal standard. IR spectra were recorded with a Shimadzu IR-408. High-resolution mass spectra were taken on a JEOL JMS-DX-300 spectrometer. Silica gel chromatography was conducted on Wako gel B5F or C-200 and alumina chromatography, activated (Wako Pure Chemical Industries LTD.).

1-Methyl-2-pyrrolidone was purchased from Wako Pure Chemical Industries Ltd. 1-Benzyl-2-piperidone,[10] 1-allyl-2-piperidone,[11] 1-benzyl-2-pyrrolidone,[12] and 1-benzyl-2-perhydroazepinone[12] were synthesized from the corresponding lactams by N-alkylation (alkyl bromide, NaH; DMF, room temperature, overnight). 3-Ethyl-1-benzyl-2-piperidone and 3-allyl-11-methyl-2-pyrrolidone[13] were prepared by α-alkylation of **2a** and **2d** (LDA, alkyl bromide, THF, -78 °C, 30 min). 1-Benzyl-5-(methoxymethyl)-2-pyrrolidone was synthesized from 5-carboxy-2-pyrrolidone by the following reactions: (i) $PhCH_2Br$, NaH; DMF, room temperature, overnight; (ii) $LiAlH_4$, THF; -20 °C, 30 min; (iii) CH_3I, NaH; DMF, room temperature, overnight.

2c: ^{1}H NMR δ 0.95 (3 H, t, J = 7 Hz), 1.2–2.2 (6 H, m), 2.8–3.3 (3 H, m), 4.57 (2 H, s), 7.25 (5 H, s); IR (neat) 1630, 740, 700 cm^{-1}.

2g: ^{1}H NMR δ 1.7–2.6 (4 H, m), 3.24 (3 H, s), 3.3–3.4 (2 H, m), 3.4–3.7 (1 H, m), 4.14 (1 H, d, J = 15 Hz), 4.90 (1 H, d. J = 15 Hz), 7.27 (5 H, s); IR (neat) 1680, 705 cm^{-1}; exact mass calcd for $C_{13}H_{17}NO_2$ 219.1259, found 219.1243.

Table II. Effect of Amines in the Synthesis of 3e

amine	yield of **3e**, %	recovery of **2e**, %
none	10	22
triethylamine	41	53
pyrrolidine	45	48
N-methylpiperadine	34	42
TMEDA	28	14
morpholine	30	70

General Procedures for the Synthesis of 2-[(*tert*-Butoxycarbonyl)methylidene]azacycloalkanes 3. To a THF (2 mL) solution of lithium diisopropyl amide (LDA), prepared from diisopropylamine (202 mg, 2.0 mmol) and a hexane solution (1.3 mL) of *n*-butyllithium (2.0 mmol), was added a THF (2 mL) solution of *tert*-butyl acetate (232 mg, 2.0 mmol) at -78 °C. After 10 min, the lactam (0.5 mmol) in THF (2 mL) and $BF_3\cdot OEt_2$ (0.4 mL) were added successively, and the mixture was allowed to react for 30 min at -78 °C. Then, pyrrolidine (1 mL) followed by water was added. The mixture was extracted with ethyl acetate, washed with water and brine, dried (Na_2SO_4), and evaporated in vacuo, and the residue was chromatographed (SiO_2, AcOEt:hexane = 1:3; alumina for **3h**) to give **3**.

3a: mp (hexane) 103–104 °C; ^{1}H NMR δ 1.42 (9 H, s), 1.5–1.9 (4 H, m), 3.0–3.4 (4 H, m), 4.39 (2 H, s), 4.64 (1 H, s), 7.0–7.4 (5 H, m); IR (KBr) 1670, 1560, 1120, 800, 730 cm^{-1}. Anal. Calcd for $C_{18}H_{25}NO_2$: C, 75.22; H, 8.77; N, 4.87. Found: C, 74.76; H, 8.72; N, 4.71.

3b: ^{1}H NMR δ 1.44 (9 H, s), 1.5–1.9 (4 H, m), 2.9–3.3 (4 H, m), 3.76 (2 H, d, J = 5 Hz), 4.55 (1 H, s), 4.9–5.3 (2 H, m), 5.5–6.1 (1 H, m); IR (neat) 1675, 1570, 1120, 800 cm^{-1}; exact mass calcd for $C_{14}H_{23}NO_2$ 237.1729, found 237.1735.

3c: ^{1}H NMR δ 1.00 (3 H, t, J = 7 Hz), 1.43 (9 H, s), 1.3–2.0 (6 H, m), 2.9–3.3 (2 H, m), 3.8–4.2 (1 H, m), 4.08 (1 H, d, J = 16 Hz), 4.46 (1 H, d, J = 16 Hz), 4.66 (1 H, s), 7.0–7.4 (5 H, m); IR (neat) 1680, 1560, 1110, 800, 730, 700 cm^{-1}; exact mass calcd for $C_{20}H_{29}NO_2$ 315.2199, found 315.2218.

3d: ^{1}H NMR δ 1.47 (9 H, s), 1.6–2.2 (2 H, m), 2.78 (3 H, s), 3.11 (2 H, t, J = 6 Hz), 3.33 (2 H, t, J = 7 Hz), 4.40 (1 H, s); IR (neat) 1680, 1600, 1135, 790 cm^{-1}; exact mass calcd for $C_{11}H_{19}NO_2$ 197.1416, found 197,1448.

3e: mp(hexane) 105–106 °C; ^{1}H NMR δ 1.46 (9 H, s), 1.7–2.2 (2 H, m), 2.78 (2 H, t, J = 6 Hz), 3.29 (2 H, t, J = 7 Hz), 4.33 (2 H, s), 4.63 (1 H, s), 7.0–7.4 (5 H, m); IR (KBr) 1680, 1590, 1120, 790, 740 cm^{-1}; exact mass calcd for $C_{17}H_{23}NO_2$ 273.1729, found 273.1729.

3f: ^{1}H NMR δ 1.47 (9 H, s), 1.6–2.6 (4 H, m), 2.77 (3 H, s), 3.1–3.6 (2 H, m), 3.6–4.0 (1 H, m), 4.37 (1 H, s), 4.8–5.2 (2 H, m), 5.4–6.1 (1 H, m); IR (neat) 1675, 1595, 1130, 790 cm^{-1}; exact mass calcd for $C_{14}H_{23}NO_2$ 237.1729, found 237.1733.

3g: ^{1}H NMR δ 1.44 (9 H, s), 1.6–2.2 (2 H, m), 3.0–3.4 (4H, m), 3.19 (3 H, s), 3.4–3.8 (1 H, m), 4.44 (2 H, s), 4.56 (1 H, s), 7.0–7.3 (5 H, m); IR (neat) 1670, 1590, 1120 cm^{-1}; exact mass calcd for $C_{19}H_{27}NO_3$ 317.1991, found 317.2006.

3h: mp (hexane) 104–105 °C; ^{1}H NMR δ 1.42 (9 H, s), 1.5–1.8 (6 H, m), 3.1–3.5 (4 H, m), 4.42 (2 H, m), 4.58 (1 H, s), 7.0–7.4 (5 H, m), IR (KBr) 1675, 1570, 1125, 800, 740 cm^{-1}; exact mass calcd for $C_{19}H_{27}NO_2$ 301.2041, found 301.2055. Piperidine was used to quench the reaction.

Reaction of Lithiated *tert*-Butyl Acetate (1) with 2b in the Presence of $TiCl_4$. To a THF (4 mL) solution of LDA (4.0 mmol), prepared as above, was added *tert*-butyl acetate (464 mg, 4.0 mmol) in THF (2 mL) at -78 °C. After 20 min, **2b** (146 mg, 1.0 mmol) in THF (2 mL) and $TiCl_4$ (0.4 mL) were added successively, and the mixture was stirred for 1.5 h at -78 °C. Then, saturated aqueous bicarbonate was added and the mixture was stirred vigorously for 1 h, extracted with ethyl acetate, washed with water and brine, dried (Na_2SO_4), and evaporated in vacuo. 1-Allyl-2,2-bis[(*tert*-butoxycarbonyl)methyl]piperidine (**4b**, 130 mg, 35%) and **3b** (69 mg, 33%) were isolated by chromatography (silica gel, ether:hexane = 1:3).

4b: ^{1}H NMR δ 1.45 (18 H, s), 1.3–1.7 (4 H, m), 1.8–2.1 (2 H, m), 2.29 (2 H, d, J = 13 Hz), 2.3–2.5 (2 H, m), 2.74 (2 H, d, J = 13 Hz), 3.06 (2 H, d, J = 5 Hz), 4.8–5.3 (2 H, m), 5.4–6.0 (1 H,

(10) "The Aldrich Library of NMR Spectra", VII, 90B.
(11) Shostakovskii, M. F.; Sidel'kovskaya, *Vysokomol. Soedin.* **1960**, *2*, 1794; *Chem. Abstr.* **1961**, *55*, 26516c.
(12) Oliveros, E.; Rivi<<accere, M.; Lattes, A. *Nouv. J. Chim.* **1979**, *3*, 739.
(13) Cresson, P. *C. R. Hebd. Seances Acad. Sci., Ser. C* **1972**, *275*, 1299.

m); IR (neat) 1720 cm^{-1}; exact mass calcd for $C_{20}H_{35}NO_4$ 353.2564, found 353.2551.

Synthesis of *tert*-Butyl 3-Oxooctanoate (6) from 1 and *N,N*-Dimethylhexanamide (5). The reaction was carried out as in the general procedure except that the reaction was quenched with triethylamine (1 mL) to give **6** (30% yield).

6: ^1H NMR δ 0.7–1.0 (3 H, m), 1.0–1.8 (6 H, m), 1.45 (9 H, s), 2.52 (2 H, t, J = 7 Hz), 3.32 (2 H, s); IR (neat) 1750–1710, 1640 cm^{-1}.

Acknowledgment. We thank Mr. Y. Nobayashi for his assistance.

Registry No. **2a**, 4783-65-7; **2b**, 28737-46-4; **2c**, 95936-18-8; **2d**, 872-50-4; **2e**, 5291-77-0; **2f**, 40296-20-6; **2g**, 95936-19-9; **2h**, 33241-96-2; **3a**, 95936-20-2; **3b**, 95936-21-3; **3c**, 95936-22-4; **3d**, 78167-70-1; **3e**, 95936-23-5; **3f**, 95936-24-6; **3g**, 95936-25-7; **3h**, 95936-26-8; **4b**, 95936-27-9; **5**, 5830-30-8; **6**, 66720-07-8; *tert*-butyl acetate, 540-88-5.

Catalytic Asymmetric Synthesis of Chiral 4-Substituted 2-Oxetanones

Hans Wynberg* and Emiel G. J. Staring

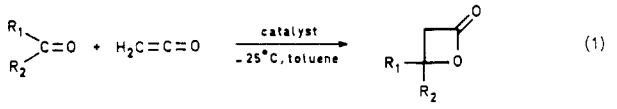
Department of Organic Chemistry, University of Groningen, Nijenborgh 16, 9747 AG Groningen, The Netherlands

Received December 7, 1984

Although 2-oxetanones are generally regarded as useful starting materials in synthesis[1] and polymerization,[2] relatively few preparations of optically active 2-oxetanones have been described. The published procedures usually consist of the ring closure of optically active β-functionalized carboxylic acid derivatives,[3] the latter obtained by resolution.[4]

The high chemical and enantiomeric yields in the C–C bond-forming reaction of ketene and chloral,[5] catalyzed by chiral tertiary amines, to produce the chiral 4-(trichloromethyl)-2-oxetanone, prompted us to investigate the reaction of other carbonyl compounds with ketene.

Table I lists the results of the reaction of some chlorinated aldehydes and ketones with ketene. The reactions were run according to the general scheme in eq 1 by



(1)

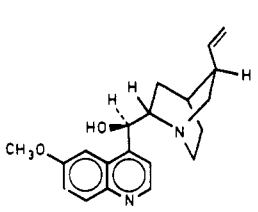
bubbling gaseous ketene through a toluene solution of the aldehyde or ketone and 1–2 mol % of the chiral catalyst at temperatures, depending on the substrate, between −25 °C and −50 °C.

Table I

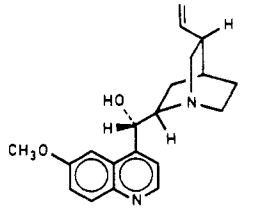
R_1	R_2	% ee *a*	% ee *b*	c.y., %
CCl_3	H	98	76	89
CCl_2H	H	45		67
CCl_2CH_3	H	91	76	95
$CCl_2CH_2CH_3$	H	89	70	87
$CCl_2C_6H_5$	H	90	68	89
CCl_3	CH_3	94	85	72
CCl_3	CH_2CH_3			1–2
CCl_3	C_6H_5			
CCl_3	C_6H_4Cl-*p*	90	65	68
CCl_3	$C_6H_4NO_2$-*p*	89	65	95

[a] Catalyst is quinidine. [b] Catalyst is quinine.

The cinchona alkaloids quinidine and quinine are used as standard catalysts. In all cases these two alkaloids give



quinine



quinidine

products with opposite signs of rotation and therefore opposite absolute configuration. Both chemical and enantiomeric yields are high in this reaction.

An important factor determining the rate of the reaction between the aldehydes and ketones seems to be the polarization of the carbonyl group. The effect of the polarization is shown nicely in the series of trichloroacetophenones. The parent compound α,α,α-trichloroacetophenone did not react with ketene to give a 2-oxetanone under a wide range of reaction conditions. If an electron-withdrawing substituent is introduced into the phenyl ring the reaction to form the 2-oxetanone does occur. Under more drastic conditions (high concentrations, excess ketene), the 2-oxetanone of *p*-chloro-α,α,α-trichloroacetophenone could be isolated in 68% chemical yield, along with some starting material. A more powerful electron-withdrawing substituent such as NO_2 leads to a quantitative conversion of the acetophenone, under standard conditions, to the 2-oxetanone. The enantiomeric purities of the two adducts are the same. No differences in the reaction rate could be observed for the chlorinated aldehydes. Monochlorinated aldehydes do not react with ketene. 1,1,1-Trichloroacetone reacts considerably slower with ketene than do the aldehydes. Excess ketene and higher concentrations are necessary to obtain satisfactory yields. The ketene adduct of 1,1,1-trichlorobutan-2-one could be isolated in trace amounts.

The absolute configuration of the 4-(trichloromethyl)-2-oxetanone, derived from ketene and chloral, was correlated to malic acid.[5] The original assignment of the *S* configuration to the 2-oxetanone yielding (*S*)-malic acid is erroneous.[6] During conversion to malic acid inversion takes place at the chiral center and the starting 2-oxetanone must therefore be of *R* configuration.[6]

To determine the absolute configuration of some of the other 2-oxetanones, their CD spectra were compared.

(1) Fujisawa, T.; Sato, T.; Kawara, T.; Noda, A.; Obinata, T. *Tetrahedron Lett.* **1980**, *21*, 2553. Sato, T.; Kawara, T.; Kawashima, M.; Fujisawa, T. *Chem. Lett.* **1980**, 571. Cannone, P.; Foscolos, G. B.; Bélanger, D. *J. Org. Chem.* **1980**, *45*, 1828. Mulzer, J.; Kerkmann, T. *J. Am. Chem. Soc.* **1980**, *102*, 3620. Normant, J. F.; Alexalis, A.; Cahiez, G. *Tetrahedron Lett.* **1980**, *21*, 935. Fujisawa, T.; Mori, T.; Kawara, T.; Sato, T. *Chem. Lett.* **1982**, 569. Sato, T.; Naruse, K.; Fujisawa, T. *Tetrahedron Lett.* **1982**, *23*, 3587.

(2) Teranishi, K.; Iida, M.; Araki, T.; Yamashita, S.; Tani, H. *Macromolecules* **1974**, *7*, 421. Iida, M.; Araki, T.; Teranishi, K.; Tani, H. *Macromolecules* **1977**, *10*, 275. Bigdeli, E.; Lenz, R. W. *Macromolecules* **1978**, *11*, 493. Chatani, Y.; Yokouchi, M.; Tadokoro, H. *Macromolecules* **1979**, *12*, 822. Grenier, D.; Prud'homme, R. E.; Leborgne, A.; Spassky, N. *J. Polym. Sci., Polym. Chem. Ed.* **1981**, *19*, 1781.

(3) Blume, R. C. *Tetrahedron Lett.* **1969**, *13*, 1047. Mulzer, J.; Pointer, A.; Chucholowski, A.; Brüntrup, G. *J. Chem. Soc., Chem. Commun.* **1979**, 52. Adam, W.; Baeza, J.; Liu, J.-C. *J. Am. Chem. Soc.* **1972**, *94*, 2000. Merger, F. *Chem. Ber.* **1968**, *101*, 2413.

(4) Sato, T.; Kawara, T.; Nishizawa, A.; Fujisawa, T. *Tetrahedron Lett.* **1980**, *21*, 3377. Grenier, D.; Prud'homme, R. E.; Leborgne, A.; Spassky, N. *J. Polym. Sci., Polym. Chem. Ed.* **1981**, *19*, 1781.

(5) Wynberg, H.; Staring E. G. J. *J. Am. Chem. Soc.* **1982**, *104*, 166.

(6) Wynberg, H.; Staring, E. G. J. *J. Chem. Soc., Chem. Commun.* **1984**, 1181.

0022-3263/85/1950-1977$01.50/0