80633-42-7; (±)-7 (R = C_6H_5CO) (isomer 1), 84731-32-8; (±)-7 (R = C_6H_5CO) (isomer 2), 84731-33-9; 9, 84731-34-0; (±)-10, 84731-35-1.

Supplementary Material Available: Full experimental details and spectral data for the synthesis of supinidine 2 from pyrrolidone 6 are available (5 pages). Ordering information is given on any current masthead page.

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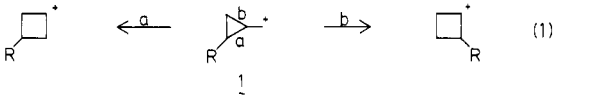
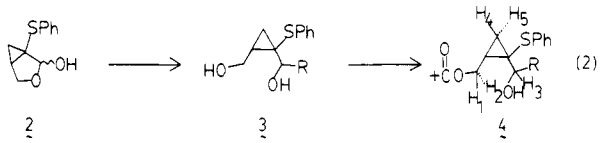
An Unusual Migratory Aptitude in a Cyclopropylcarbinyl–Cyclobutyl Rearrangement. Synthesis of 2,4-Disubstituted Cyclobutanones

Summary: The inductive effect of an (acyloxy)methyl substituent encourages migration of the *less* substituted carbon in the Grignard adducts of (*E*)-2-(hydroxymethyl)-1-(phenylthio)cyclopropane-1-carboxaldehyde.

Sir: The utility of cyclobutanones as synthetic intermediates surges as their accessibility increases.[1,2] [2 + 2] Cycloadditions of ketenes[2,3] and ketene iminium salts[4] provide ready access to 2,3-disubstituted cyclobutanones.[5] Sprioannulation with diphenylsulfonium cyclopropylide[6] and 1-lithiocyclopropyl phenyl sulfide[7] as well as the oxygen[8] and selenium[9] analogues of the latter generates 1,1-disubstituted cyclobutanones. We record a facile approach to 2,4-disubstituted cyclobutanones that derives from an unusual migratory aptitude in a cyclopropylcarbinyl–cyclobutyl rearrangement in which a remote substituent diverts the normally preferred migration of the more substituted carbon in 1 (i.e., bond a)[10] to the less substituted carbon (i.e., bond b) (see eq 1). The magnitude as well as the direction of this reversal is particularly surprising considering the normal migratory aptitudes reported for such reactions.[10]



Addition of Grignard reagents to the lactol 2[11] in THF at 0 °C provides the diol 3 in virtually quantitative yield. Chemoselective acylation of the primary alcohol occurs smoothly with pivaloyl chloride (DMAP, C_5H_5N, CH_2Cl_2, 0 °C) to give the hydroxypivalates 4 in 70–82% overall yield from 2 (see Table I and eq 2). The IR spectra



showed characteristic absorptions at 3580 ± 20 cm^{-1} for the hydroxyl group and 1725 ± 5 cm^{-1} for the ester carbonyl group. The 270-MHz NMR spectra showed H_1 and H_2 as doublet of doublets at δ 4.48 ± 0.12 (J = 12.1, 5.5 ± 0.25) and δ 4.00 ± 0.25 (J = 12.1, 9.9 ± 0.6 Hz), H_3 as a multiplet at δ 3.64 ± 0.36 (except for 4, R = Ph, at δ 4.71), H_4 as a multiplet or doublet of doublets at δ 1.30 ± 0.06 (J = 9.4 ± 0.2, 5.4 ± 0.3 Hz), and H_5 as a triplet at δ 0.94 ± 0.18 (J = 5.8 ± 0.4 Hz). In each case 4 existed as a 1:1 to 2:1 diastereomeric mixture, which normally was not separated.

Table I. Synthesis of 2,4-Disubstituted Cyclobutanones

entry	Grignard reagent	4,[a] R, yield, %	5,[a] yield, %
1	CH_3MgBr	CH_3, 76	48
2	n-C_4H_9MgBr	n-C_4H_9, 77	59
3	i-C_3H_7MgBr	i-C_3H_7, 70	46
4	CH_2=$CHCH_2CH_2MgBr$	CH_2=$CHCH_2CH_2$, 75	38
5	$PhCH_2CH_2MgBr$	$PhCH_2CH_2$, 82	55
6	CH_2=$CHMgBr$	CH_2=CH, 70	ND[b]
7	PhMgBr	Ph, 74	ND[b]

[a] All new compounds have been fully characterized by spectral means and elemental composition determined by high-resolution mass spectroscopy and/or combustion analysis. [b] ND = not determined.

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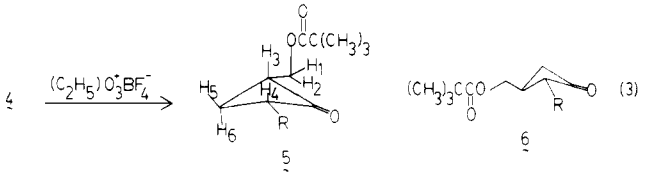
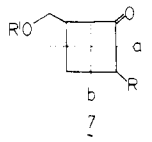
Rearrangement to cyclobutanones proved especially troublesome. Standard conditions for the parent cyclopropyl system[7] such as aqueous fluoroboric acid in water–ether, *p*-toluenesulfonic acid in moist benzene, and stannic chloride either returned starting material or produced complex mixtures. On the other hand, the fluoroborate salts of trimethyloxonium, triethyloxonium, or dimethoxycarbenium ions (room temperature in methylene chloride for 2 h) effected the rearrangements to the stereo- and regiochemically homogeneous cyclobutanones (IR 1775 cm^{-1}) as outlined in the table and eq 3. For R = CH_2=CH and Ph, analysis of the crude reaction mixture showed the presence of the cyclobutanones, but isolation of the pure products was not possible.

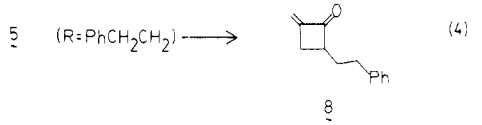


(3)

Two regioisomeric cyclobutanones, **5** or **6**, are possible. Our previous work with the methyl-substituted cyclopropylphenyl sulfide and diphenylsulfonium cyclopropylide[6,7,8,12] as well as earlier work on the cyclopropylcarbinyl–cyclobutyl rearrangement led us to expect **6** as the major product; however, the NMR and mass spectra were inconsistent with this assignment. The proton NMR assignments for **5** (R = *n*-C_4H_9) follow: δ 4.27 (dd, J = 11.0, 5.0 Hz, 1 H) and 4.21 (dd, J = 11.0, 4.8 Hz, 1 H), for H_1 and H_2, 3.52 (m, 1 H), for H_3, 3.25 (m, 1 H), for H_4, 2.09 (ddd, J = 11.5, 10.0, 6.0 Hz, 1 H), and 1.87 (ddd, J = 11.5, 10.0, 6.0 Hz, 1 H) for H_5 and H_6. Spin decoupling confirms these assignments. Particularly noteworthy, irradiation of the pivaloxymethylene protons reveals that H_3 shows vicinal coupling only to H_5 and H_6 with only a small long-range coupling to H_4. The mass spectra of cyclobutanones exhibit peaks corresponding to the two possible [2 + 2] cycloreversions as in **7**. For **5** (R = C_4H_9) these peaks appear at m/e 142 and 98 for scission a and at m/e 156 and 84 for scission b. These observations are inconsistent with **6**.



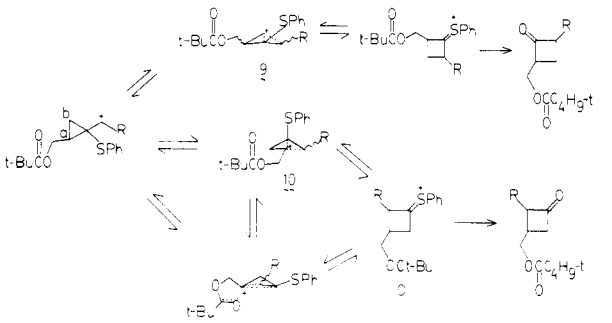
Chemical evidence in support of this regiochemical assignment derived from a facile base-catalyzed elimination ($NaOCH_3$, CH_3OH, room temperature, 30 min) to the 2-methylenecyclobutanone **8**. This observation not only



(4)

confirms the 2,4-disubstitution but also demonstrates the utility of this methodology for the synthesis of methylenecyclobutanones, potentially very exciting building blocks.[13]

Scheme I



^{13}C NMR, TLC, and GLC analyses confirm the stereo- and regiochemical homogeneity of the cyclobutanones. The *Z* stereochemistry is assigned on the known thermodynamic preference of 2,4-disubstituted cyclobutanones for the *Z* isomer.[14]

The regioselectivity of the rearrangement is most interesting. To verify that the stereochemistry of the diastereomeric hydroxypivalates did not determine the regiochemistry, we subjected each diastereomer of **4**, *R* = *n*-C_4H_9, separately to the reaction conditions. The same cyclobutanone derived from these experiments as from rearrangement of the diastereomeric mixture.

Two factors are important in determining the outcome of carbonium ion rearrangements: (1) the ability of the migrating group to stabilize a positive charge (migratory aptitude) and (2) the stability of the newly formed positive charge. The importance of each of these factors for the rearrangement of carbinols **4** can be seen by examining some of the possible intermediates or transition states (see Scheme I). As either carbon a or carbon b begins to migrate, positive charge develops at that carbon atom; thus, the electronic nature of this carbon becomes an important factor in determining its migratory aptitude. For the migration of carbon a, additional delocalization is possible via anchimeric assistance of the type shown.[15] However, the inductive effect of the pivaloxymethyl substituent diminishes the importance of any such overlap and favors migration of the less substituted carbon b.[16] Further, the $+\sigma_I$ for an (acyloxy)methyl group[17] leads to the conclusion that **10** is destabilized compared to **9** due to the proximity of the pivaloxymethyl group to the migrating carbon. This latter factor appears to dominate in determining the migratory aptitudes. The utility of such remote inductive effects for control of selectivity would appear to be greater than is generally assumed[18] and provides another dimension for selectivity in the design of new conjunctive reagents.

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Acknowledgment. We thank the National Science Foundation for their generous support of our programs.

Registry No. 2, 80398-85-2; 3 (R = CH_3), 84851-70-7; 3 (R = n-C_4H_9), 84851-71-8; 3 (R = i-C_3H_7), 84851-72-9; 3 (R = $CH_2CH_2CH{=}CH_2$), 84851-73-0; 3 (R = CH_2CH_2Ph), 84851-74-1; 3 (R = $CH{=}CH_2$), 84851-75-2; 3 (R = Ph), 84851-76-3; 4 (R = CH_3) (isomer 1), 84851-77-4; 4 (R = CH_3) (isomer 2), 84894-10-0; 4 (R = n-C_4H_9) (isomer 1), 84851-78-5; 4 (R = n-C_4H_9) (isomer 2), 84894-11-1; 4 (R = i-C_3H_7) (isomer 1), 84851-79-6; 4 (R = i-C_3H_7) (isomer 2), 84894-12-2; 4 (R = $CH_2CH_2CH{=}CH_2$) (isomer 1), 84851-80-9; 4 (R = $CH_2CH_2CH{=}CH_2$) (isomer 2), 84894-13-3; 4 (R = CH_2CH_2Ph) (isomer 1), 84851-81-0; 4 (R = CH_2CH_2Ph) (isomer 2), 84894-14-4; 4 (R = $CH{=}CH_2$) (isomer 1), 84851-82-1; 4 (R = $CH{=}CH_2$) (isomer 2), 84894-15-5; 4 (R = Ph) (isomer 1), 84851-83-2; 4 (R = Ph) (isomer 2), 84894-16-6; 5 (R = CH_3), 84851-84-3; 5 (R = n-C_4H_9), 84851-85-4; 5 (R = i-C_3H_7), 84851-86-5; 5 (R = $CH_2CH_2CH{=}CH_2$), 84851-87-6; 5 (R = CH_2CH_2Ph), 84851-88-7; 5 (R = $CH = CH_2$), 84851-89-8; 5 (R = Ph), 84851-90-1; 8, 84851-91-2; CH_3Br, 74-83-9; n-C_4H_9Br, 109-65-9; i-C_3H_7Br, 75-26-3; $CH_2{=}CHCH_2CH_2Br$, 5162-44-7; $PhCH_2CH_2Br$, 103-63-9; $CH_2{=}CHBr$, 593-60-2; PhBr, 108-86-1; pivaloyl chloride, 3282-30-2.

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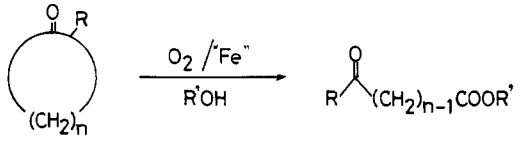
Received November 29, 1982

Ferric Salt Catalyzed Oxygenation of Cycloalkanones to Oxo Esters by Molecular Oxygen

Summary: Ferric salts catalyzed the regiospecific oxygenation of cycloalkanones by molecular oxygen to give oxo esters in the presence of aliphatic alcohol under mild conditions.

Sir: Transition-metal-catalyzed oxygenation by molecular oxygen has received much attention from a synthetic point of view as well as in connection with the oxygenation in biological systems.[1] However, there have been few examples of efficient iron-catalyzed oxygenations by molecular oxygen.[2]

We now report that regiospecific oxidative ring-opening of cycloalkanones[3] to oxo esters by molecular oxygen in the presence of aliphatic alcohol is catalyzed by simple ferric salts, which seems to activate substrate and molecular oxygen simultaneously (eq 1).



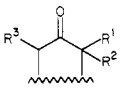
(1)

Table I. $FeCl_3$-Catalyzed Oxygenation of 1 with[a] Some Alcohols as Nucleophiles

ROH	yield, %
MeOH	93
EtOH	79
$PhCH_2OH$	80
t-$BuCH_2OH$	63
i-PrOH	73
t-BuOH	42
H_2O	86

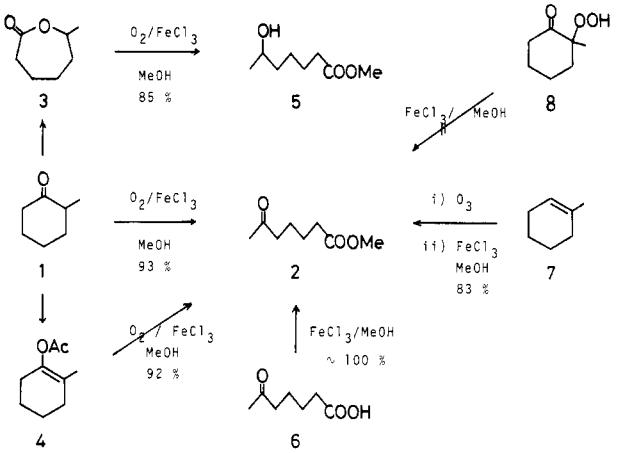
[a] Carried out using 10 equiv of ROH in benzene at 60 °C under oxygen atmosphere (1 atm).

Table II. Substituent Effect on the Oxygenation of Substituted Cyclohexanones[a]

run		conv, %	yield, %
1	$R^1 = R^2 = R^3 = H$	14	66[b]
2	R^1 = Me, $R^2 = R^3 = H$	100	93
3	R^1 = Et, $R^2 = R^3 = H$	100	81
4	R^1 = t-Bu, $R^2 = R^3 = H$	28	65
5	R^1 = OMe, $R^2 = R^3 = H$	5	0
6	$R^1 = CF_3$, $R^2 = R^3 = H$	5	0
7	R^1 = Br, $R^2 = R^3 = H$	46	complex
8	R^1 = Me, R^2 = H, R^3 = Me	60	63
9	R^1 = Me, R^2 = Cl, R^3 = Me	35	0[c]

[a] Carried out in methanol with $FeCl_3$. [b] A mixture of methyl 6-oxohexanoate and its dimethyl acetal (1:2). [c] Dehydrochlorination yielding 2,6-dimethylcyclohexenone was observed.

Chart I



Treatment of a benzene solution of 2-methylcyclohexanone (1) (0.2 M) including 10 equiv of methanol and a catalytic amount of $FeCl_3$ (2–5 mol %) at 60 °C under oxygen atmosphere (1 atm) for 20 h gave methyl 6-oxoheptanoate (2) in 93% yield. $FeCl_3$ can be replaced by other ferric salts such as $Fe(NO_3)_3$, $FeBr_3$, and $K_3Fe(CN_6H^+$ but not by ferric organometalic complexes such as $Fe(acac)_3$.[6] Ferrous salts also showed catalytic activity, though less than that of ferric salts ($FeCl_2$, 24% yield).[7] Methanol could be replaced by aliphatic alcohols as summarized in Table I. Not only primary and secondary but tertiary and sterically hindered alcohols could form an

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(2) (a) Ito, S.; Inoue, K.; Matsumoto, M. *J. Am. Chem. Soc.* **1982**, *104*, 6450. (b) Paulson, D. R.; Ullman, R.; Sloane, R. B.; Closs, G. L. *J. Chem. Soc., Chem. Commun.* **1974**, 186.

(3) Autoxidation of cycloalkanones is known to be catalyzed by base[4] as well as certain transition metals such as Co and Mn.[5] However, in general, products were not oxo esters but dicarboxylic acids.

(4) For reviews, see: (a) Russell, G. A.; Bemis, A. G.; Geels, E. J.; Janzen, E. G.; Moye, A. J. *Adv. Chem. Ser.* **1968**, *No. 75*, 174. (b) Sosnovsky, G.; Zaret, E. H. "Organic Peroxides"; Swern, D., Ed.; Wiley: New York, 1970; Vol. 1, p 517.

(5) (a) Onopchenko, A.; Schulz, J. G. D. *J. Org. Chem.* **1973**, *38*, 3729. (b) Prengle, H. W.; Barona, N. *Hydrocarbon Process.* **1970**, *49*, 106.

(6) Although the reason is now ambiguous, $Fe(Acac)_3$ seems not to act as a competent Lewis acid for this oxidation.

(7) Hammond showed that LiCl accelarated the oxidaton of $FeCl_2$ by molecular oxygen in methanol.[8] Nevertheless, LiCl did not practically improve the $FeCl_2$-catalyzed oxidation of 1 in methanol.

(8) Hammond, G. S.; Wu, C. S. *Adv. Chem. Ser.* **1968**, *No. 77*, 186.

0022-3263/83/1948-1133$01.50/0