29-0; 15, 35639-11-3; 2-methyl-3-phenylcyclohexanone, 18018-02-5; 2-methyl-3-phenylcyclohexanone (DNP), 18018-03-6.

**Acknowledgments.**—We are grateful for financial assistance to R. R. M. through National Institutes of Health Biomedical Sciences Support Grant No. FR-7059-05. The research was supported directly by grants from the Rutgers Research Council and in part by a grant for peripheral work from the Petroleum Research Fund of the American Chemical Society. We thank all these sources for their aid and express our appreciation to Dr. Gree Loober Spoog for helpful discussions.

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# Decarboxylation of Halogenated 2-Oxetanones

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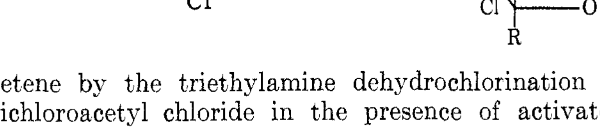
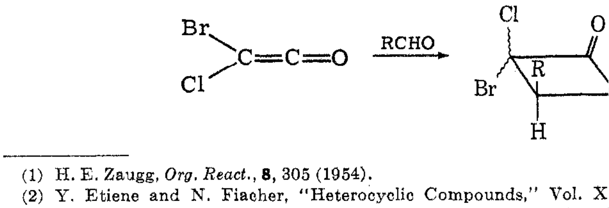
Halogenated 2-oxetanones are less susceptible to decarboxylation than other 2-oxetanones. A trichloromethyl substituent in the 4 position of the 2-oxetanone ring severely inhibits decarboxylation. The decarboxylation of 2-oxetanones derived from the cycloaddition of halogenated ketenes and chloral over an electrically heated wire produces halogenated allenes. This method provides a useful synthesis for trichloromethylallenes.

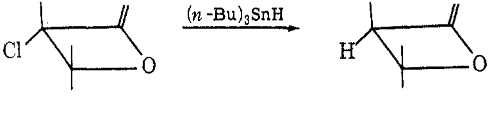
It is well known that 2-oxetanones are quite susceptible to thermal decarboxylations to yield olefinic compounds.[1,2] However, the effect of substituents such as a halogen on the rate of thermal decarboxylations is relatively unknown. In our investigations concerning the cycloaddition of halogenated ketenes and carbonyl compounds to produce 2-oxetanones we have prepared a number of halogenated 2-oxetanones. It was of interest to study the decarboxylation of these compounds and determine the effect of halogen and other electronegative substituents on the thermal stability of the 2-oxetanones. Consequently, the purpose of this paper is to relate the results of our study on the decarboxylation of halogenated 2-oxetanones. A preliminary report describing a novel method for the preparation of some trichloromethylallenes has appeared.[3]

**Preparation of Halogenated 2-Oxetanones.**—We have recently reported the cycloaddition of dichloroketene with several simple ketones to produce 3,3-dichloro-2-oxetanones.[4] The generation of dichloroketene by the triethylamine dehydrochlorination of dichloroacetyl chloride in the presence of activated carbonyl compounds also yields 2-oxetanones.[5] This method has been applied to the cycloaddition of bromochloroketene with certain aldehydes to produce 3-bromo-3-chloro-2-oxetanones.





The 3,3-dichloro-2-oxetanones may be selectively reduced with tri-*n*-butyltin hydride to the corresponding monochloro-2-oxetanones. The reduction may also be effected to produce the nonhalogenated-2-oxetanones.

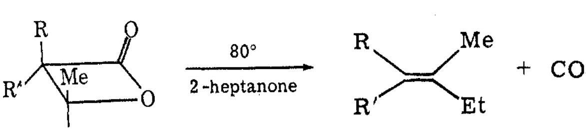


Alkylchloroketenes also undergo *in situ* cycloadditions but only with activated carbonyl compounds such as chloral to yield the expected 2-oxetanones. We have also previously described the cycloaddition of chloroketene with chloral at room temperature to yield both *cis*- and *trans*-2-oxetanones.[6] We have since found that conducting this reaction at −78° and allowing warming to room temperature produces only the trans isomer.

**Decarboxylations.**—The effect of halogen in the 3 position upon the rate of decarboxylation was easily determined by comparing the rates of decarboxylation of the compounds in Table I. The relative rates represent the time required for 50% decarboxylation. The decarboxylation was measured by observing the disappearance of the carbonyl band in the infrared using the carbonyl band of the solvent, 2-heptanone, as an internal standard.

TABLE I

EFFECT OF CHLORO SUBSTITUENT IN 3 POSITION ON RATE OF DECARBOXYLATION



| Compd | R | R′ | Relative rate |
|---|---|---|---|
| I | H | H | >100 |
| II | H | Cl | 6 |
| III | Cl | Cl | 1 |

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[1] H. E. Zaugg, *Org. React.*, **8**, 305 (1954).

[2] Y. Etiene and N. Fiacher, "Heterocyclic Compounds," Vol. XIX, Part 2, A. Weissberger Ed., Interscience, New York, N. Y., 1964 pp 729–880.

[3] W. T. Brady and A. D. Patel, *J. Chem. Soc. D*, 1642 (1971).

[4] W. T. Brady and A. D. Patel, *J. Heterocycl. Chem.*, **8**, 739 (1971).

[5] D. Borrmann and R. Wegler, *Chem. Ber.*, **102**, 64 (1969).

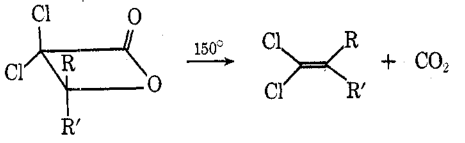
[6] W. T. Brady and L. Smith, *J. Org. Chem.*, **36**, 1637 (1971).

A further comparison of the effect of chloro substituents in the 3 position was observed when 3,3-dichloro-4-ethyl-4-methyl-2-oxetanone, III, did not decarboxylate in water up to 50° whereas isovalerolactone (4,4-dimethyl-2-oxetanone) readily decarboxylated at room temperature.[7]

Similarly, the effect of a chloromethyl substituent in the 4 position on the rate of decarboxylation can be seen in Table II. The relative rates represent the time required for completion of 50% of the reaction. The rate of decarboxylation was measured by observing the disappearance of 2-oxetanones and appearance of olefin by vpc.

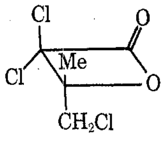
TABLE II

EFFECT OF CHLOROMETHYL SUBSTITUENT IN 4 POSITION ON RATE OF DECARBOXYLATION

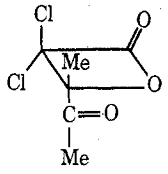


| Compd | R | R' | Relative rate |
|---|---|---|---|
| III | Me | Et | 116 |
| IV | Me | $CH_2Cl$ | 17 |
| V | $CH_2Cl$ | $CH_2Cl$ | 1 |

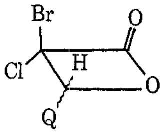
The rate of decarboxylation of the 2-oxetanones derived from dichloroketene and chloroacetone and dimethylglyoxal, IV and VI, was compared at 170° and the rates were approximately the same. Presumably, the attainment of a conjugated system in VI upon decarboxylation serves as a driving force for the decarboxylation. This ease of decarboxylation was also observed when the 2-oxetanones obtained from bromochloroketene and benzaldehyde and isobutyraldehyde, VII and VIII, were prepared. Compound VII could not be distilled under reduced pressure as decarboxylation occurred at ~100° to yield the corresponding styrene derivative, while VIII could be isolated and did not appreciably decarboxylate prior to ~160°.[8]



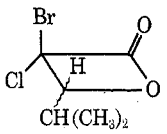
IV



VI

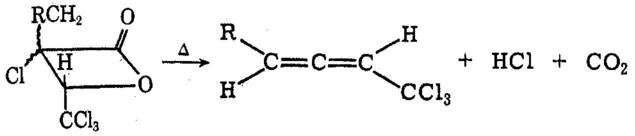


VII



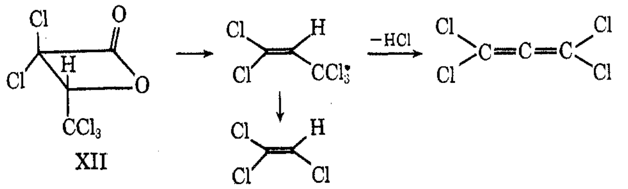
VIII

The decarboxylation of the 2-oxetanones derived from methylchloroketene and chloral and *p*-chlorobenzaldehyde and *sym*-dichlorotetrafluoroacetone did not take place up to 250°. However, decarboxylation of the cycloadducts of alkylchloroketene and chloral did take place over an electrically heated wire but this elimination was accompained by a dehydrochlorination to yield trichloromethylallenes in 40–60% yields. No evidence of the olefin was found in any system. A consideration of the order of the two elimination steps suggests that the dehydrochlorination preceded the decarboxylation.



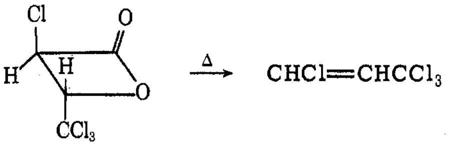
IX, R = H
X, R = Me
XI, R = Et

The 2-oxetanone obtained from dichloroketene and chloral, XII, was likewise very resistant to decarboxylation. However, refluxing XII over an electrically heated wire produced a mixture of products including the expected olefin, 1,1,1,3,3-pentachloropropene, the allene, the allene dimer, and trichloroethylene. The perchloroallene was observed in a Dry Ice–acetone trap at −78° as evidenced by a band in the infrared at 1962 cm$^{-1}$;[9] this band disappeared upon warming to room temperature but the allene dimer was isolated.[10]



The olefin, 1,1,1,3,3-pentachloropropene, dehydrochlorinates under the reaction conditions to yield perchloroallene which dimerizes upon warming to room temperature.

The cycloadduct obtained from chloroketene and chloral, XIII, was also quite resistant to decarboxylation. However, decarboxylation did occur as above but the corresponding olefin, 1,1,1,3-tetrachloroethene, was produced in 80% yield. A trace of allene (trichloroallene) was observed in the cold trap at −78° as evidenced by a band in the infrared at 1962 cm$^{-1}$.[11] However, this was a much weaker band than in the previous system. This decarboxylation was conducted on the *trans*-2-oxetanone and an equal mixture of the two isomeric olefins was obtained.



XIII

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(7) T. L. Gresham, J. E. Jansen, F. W. Shaver, and W. L. Beears, *J. Amer. Chem. Soc.*, **76**, 486 (1954).

(8) Both isomers of VII and VIII were produced in the cycloaddition reaction in approximately equal amounts and decarboxylation yielded both isomers of the olefin.

(9) A. Roedig, F. Bischoff, B. Heinrich, and G. Markl, *Justus Liebigs Ann. Chem.*, **670**, 8 (1963).

(10) A. Roedig and P. Bernemann, *ibid.*, **600**, 1 (1956).

(11) A. Roedig and N. Detzer, *Angew. Chem., Int. Ed. Engl.*, **7**, 471 (1968).

## Conclusions

It is very apparent from our studies that any electronegative substituent on the 2-oxetanone ring decreases the rate of decarboxylation. However, if conjugation results from decarboxylation, this seems to serve as a driving force for the elimination and decarboxylation occurs more readily than expected. The presence of a trichloromethyl substituent on the 4 position of the 2-oxetanone ring severely inhibits decarboxylation. This is perhaps an indication of the mechanism of the elimination reaction and suggests some charge separation whereby the 4 carbon assumes some positive character. Decarboxylation of the alkylchloroketene chloral adducts over an electrically heated wire provides a useful synthesis for these exotic trichloromethylallenes.

## Experimental Section

Nuclear magnetic resonance (nmr) spectra were recorded on a Jeolco Minimar 60-Mcps or Jeolco NMR PS 100-Mcps spectrometer. An F & M Scientific Model 700 gas chromatograph with columns of 10% SE-30 on Chromosorb W (DMSC) 60–80 mesh was used for analytical purposes.

Ether and hexane were distilled from sodium immediately prior to use as solvents. The acid halides were commercially available or prepared from the corresponding acid and thionyl chloride except for bromochloroacetyl chloride which was prepared by the general procedure of Crompton and Vanderstichele.[12]

The preparation of the 2-oxetanones derived from dichloroketene and methylethyl ketone, chloroacetone and *sym*-dichloroacetone, as well as the adduct from methylchloroketene and chloral have been described by us.[4] The 2-oxetanone from dichloroketene and chloral has been reported by Borrmann and Wegler.[5]

**3-Chloro-4-ethyl-4-methyl-2-oxetanone (II).**—A solution of 0.05 mol of freshly distilled tri-*n*-butyltin hydride in hexane was slowly added to a stirred solution of 0.05 mol of III in hexane. The reaction solution was kept cool by cold water. After it stirred for 2 hr, the hexane was evaporated under reduced pressure and the residue distilled at 40–43° (1.5 mm) in quantitative yield. Both cis and trans isomers were obtained in approximately equal amounts: ir 1865 cm$^{-1}$ ($C{=}O$); nmr ($CCl_4$) $\delta$ 1.0 (t, 3 H), 1.5 (2 s of cis and trans isomers, 3 H), 1.80 (m, 2 H), and 4.92 (s, 1 H).

*Anal.* Calcd for $C_6H_9ClO_2$: C, 48.48; H, 6.66. Found: C, 48.36; H, 6.53.

**4-Ethyl-4-methyl-2-oxetanone (I).**—A solution of 0.05 mol of tri-*n*-butyltin hydride in hexane was added to a solution of 0.025 mol of III and 0.1 g of azobisisobutyronitrile in hexane. The reaction solution was cooled by cold water. After it stirred for 2 hr, the solvent was removed under reduced pressure and I distilled at 60–65° (10 mm) [lit.[13] 60° (10 mm)].[13]

**3,3-Dichloro-4-acetyl-4-methyl-2-oxetanone (VI).**—A solution of 1.5 mol of triethylamine in 300 ml of hexane containing 1.5 mol of dimethylglyoxal was stirred while a solution of 1 mol of dichloroacetyl chloride in hexane was added at room temperature. After completion of the addition, the reaction mixture was stirred 4 hr and then filtered. The solvent was removed under reduced pressure and VI distilled at 85° (3 mm) (40% yield): ir 1870 cm$^{-1}$ ($C{=}O$); nmr ($CCl_4$) $\delta$ 1.85 (s, 3 H) and 2.4 (s, 3 H).

*Anal.* Calcd for $C_6H_6Cl_2O_3$: C, 36.58; H, 3.08. Found: C, 36.33; H, 2.92.

**3-Bromo-3-chloro-4-phenyl-2-oxetanone (VII).**—The same procedure was employed as described above for VI except bromochloroacetyl chloride was added to a solution containing benzaldehyde. Prior to vacuum distillation the residue revealed a band in the ir at 1870 cm$^{-1}$. The nmr spectrum of this residue after complete removal of the solvent revealed a singlet at $\delta$ 5.66 and a singlet at 6.0 in equal amounts for the methinyl hydrogens in both isomers. Distillation resulted in decarboxylation and $\beta$-bromo-$\beta$-chlorostyrene was obtained in 35% overall yield at 80° at 0.5 mm: ir 1600 cm$^{-1}$; nmr ($CCl_4$) $\delta$ 6.95 and 7.05 (2 s of cis and trans isomers, 1 H) and 7.25 (m, 5 H).

*Anal.* Calcd for $C_8H_6BrCl$: C, 44.14; H, 2.75. Found: C, 44.18; H, 2.61.

**3-Bromo-3-chloro-4-isopropyl-2-oxetanone (VIII).**—The same procedure was employed as described above for cycloaddition with benzaldehyde. Vacuum distillation afforded a 20% yield of two isomers in equal amounts at 60° (0.5 mm): ir 1872 cm$^{-1}$ ($C{=}O$); nmr ($CCl_4$) $\delta$ 1.15 (d, 6 H), 1.93 (m, 1 H), and 4.26 and 4.40 (2 d for cis and trans isomers, 1 H).

*Anal.* Calcd for $C_6H_8BrClO_2$: C, 31.63; H, 3.43. Found: C, 31.23; H, 3.41.

**3-Chloro-3-ethyl-4-trichloromethyl-2-oxetanone (X).**—The same procedure was employed as described for IX.[4] This 2-oxetanone was obtained at 72–75° (0.5 mm) in 50% yield: ir 1875 cm$^{-1}$ ($C{=}O$); nmr ($CCl_4$) $\delta$ 1.23 (t, 3 H), 2.5 (m, 2 H), and 4.82 and 5.18 (2 s, 1 H).

*Anal.* Calcd for $C_6H_6Cl_4O_2$: C, 28.57; H, 2.38. Found: C, 28.52; H, 2.33.

**3-Chloro-3-*n*-propyl-4-trichloromethyl-2-oxetanone (XI).**—The same procedure was employed as described for IX.[4] This 2-oxetanone was obtained at 72–78° (0.5 mm) in 45% yield: ir 1875 cm$^{-1}$ ($C{=}O$); nmr ($CCl_4$) $\delta$ 1.0 (m, 3 H), 1.7 (m, 4 H), and 4.82 and 5.18 (2 s, 1 H).

*Anal.* Calcd for $C_7H_8Cl_4O_2$: C, 31.57; H, 3.00. Found: C, 31.61; H, 3.05.

***trans*-3-Chloro-4-trichloromethyl-2-oxetanone (XIII).**—The *in situ* cycloaddition of chloroketene and chloral at room temperature produces both cis and trans isomers as we have previously described.[6] The following procedure produces only the trans isomer as evidenced by the nmr coupling constant ($J$ = 3 cps) of the methinyl protons.

A solution of 1 mol of triethylamine in 200 ml of hexane was cooled to −78° and then 1 mol of chloroacetyl chloride added dropwise. After the addition was complete, the reaction mixture was stirred at this temperature for 15–20 min and then 1.5 mol of chloral was added. After the mixture warmed to room temperature, the salt was removed by filtration, the filtrate concentrated, and the residue vacuum distilled to yield only *trans*-3-chloro-4-trichloromethyl-2-oxetanone.

**Decarboxylation of I, II and III.**—Three solutions of 1.5 g of 2-heptanone containing 1.5 g each of I, II, and III were decarboxylated at 80° in an oil bath. The rate of decarboxylation was determined by observing the disappearance of the 2-oxetanone carbonyl band in the ir at 5.4 m*M*. The carbonyl band of 2-heptanone at 5.85 m*M* was used as an internal standard. The relative rates were 1:6:>100 for III, II, and I, respectively.

**Decarboxylation of III, IV, and V.**—A 3-g portion of each of the 2-oxetanones, III, IV, and V, was thermally decarboxylated at 150° in an oil bath. The rate of decarboxylation was determined by vpc analysis by observing the disappearance of 2-oxetanone and appearance of the olefin. The relative rates were found to be 1:17:116 for V, IV, and III, respectively.

**Decarboxylation of IX. Trichloromethylallene.**—A 24-g (0.1 mol) portion of IX was pyrolyzed in a ketene generator at 1.5–2 mm as the 2-oxetanone was slowly refluxed over the electrically heated red hot filament. After about 2 hr, trichloromethylallene was isolated from a Dry Ice–acetone trap in the system. Distillation afforded 9.4 g (60%) of the allene at 80–82°: ir 1601 (vs) and 1965 cm$^{-1}$ (w); nmr ($CCl_4$) $\delta$ 5.36 (d, 2 H) and 6.03 (s, 1 H).

*Anal.* Calcd for $C_4H_3Cl_3$: C, 30.47; H, 1.94; mol wt, 156. Found: C, 30.32; H, 1.91; mol wt, 156 (by mass spectrometry).

**Decarboxylation of X. *1,1,1-Trichloro-2,3-pentadiene.***—The pyrolysis was effected as described above for IX and the allene was obtained in a 50% yield. Purification was accomplished by preparative vpc: ir 1600 cm$^{-1}$; nmr ($CCl_4$) $\delta$ 1.75 (d, 3 H), 6.12 (q, 1 H), and 6.32 (s, 1 H).

*Anal.* Calcd for $C_5H_5Cl_3$: C, 34.98; H, 2.90; mol wt, 170. Found: C, 35.16; H, 2.79; mol wt, 170 (by mass spectrometry).

**Decarboxylation of XI. *1,1,1-Trichloro-2,3-hexadiene.***—The same procedure was employed as described above for IX and the allene was obtained in 40% yield and purified by preparative vpc: ir 1600 cm$^{-1}$; nmr ($CCl_4$) $\delta$ 1.0 (t, 3 H), 2.13 (m, 2 H), 6.08 (t, 1 H), and 6.22 (s, 1 H).

*Anal.* Calcd for $C_6H_7Cl_3$: C, 38.81; H, 3.77; mol wt, 184. Found: C, 38.66; H, 3.65; mol wt, 184 (by mass spectrometry).

**Decarboxylation of XII.**—A 26-g (0.1 mol) portion of XII was pyrolyzed under the same conditions as described above. The products of the pyrolysis were collected in a Dry Ice–acetone trap at −78°. There was a strong band in the ir at 1962 cm$^{-1}$ at low temperature which disappeared upon warming to room tempera-

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[12] H. Crompton and P. Vanderstichele, *J. Chem. Soc.*, **117**, 691 (1920).

[13] H. J. Hagemeyer, *Ind. Eng. Chem.*, **41**, 765 (1949).

ture. Distillation afforded 7.5 g (35%) of 1,1,1,3,3-pentachloropropene, 1.7 g (13%) of trichloroethylene, and 1.8 g (11%) of perchloroallene dimer which crystallized from the residue of the distillation, mp 90–91° (lit.[9] 90–91°). The ir spectrum was identical with that reported in the literature.[9]

Pyrolysis of 5 g of 1,1,1,3,3-pentachloropropene under identical conditions at 25-mm pressure produced products in the Dry Ice–acetone trap which gave a band in the ir at 1962 cm$^{-1}$ at low temperature.

**Decarboxylation of XIII.**—A 20-g (0.09 mol) portion of XIII was pyrolyzed in a ketene generator under reduced pressure as described above. A 13-g (80%) portion of 1,1,1,3-tetrachloropropene was isolated from the Dry Ice trap. This olefin was approximately an equal amount of cis and trans isomers as evidenced by the nmr coupling constants for the vinyl protons.[14]

**Registry No.**—I, 4288-03-3; *cis*-II, 35589-64-1; *trans*-II, 35621-77-3; III, 34624-15-2; IV, 34499-08-6; V, 34499-11-1; VI, 35621-81-9; *cis*-VII, 35621-82-0; *trans*-VII, 35621-83-1; *cis*-VIII, 35621-84-2; *trans*-VIII, 35621-85-3; *cis*-X, 35621-86-4; *trans*-X, 35621-87-5; *cis*-XI, 35621-88-6; *trans*-XI, 35621-89-7; *trans*-XIII, 28186-54-1; *cis*-β-bromo-β-chlorostyrene, 35621-91-1; *trans*-β-bromo-β-chlorostyrene, 35621-92-2; trichloromethylallene, 34819-62-0; 1,1,1-trichloro-2,3-pentadiene, 34819-63-1; 1,1,1-trichloro-2,3-hexadiene, 34819-64-2.

**Acknowledgments.**—The authors would like to express their appreciation to the Robert A. Welch Foundation and the North Texas State University Faculty Research Fund for their generous support of this work.

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[14] R. Fields, R. N. Haszeldine, and D. Peter, *J. Chem. Soc. C*, 165 (1969).

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# A Novel Variant of the Favorskii Reaction[1]

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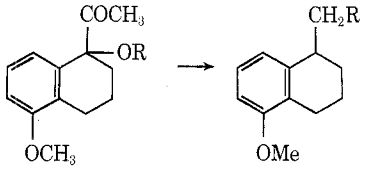
*Received May 2, 1972*

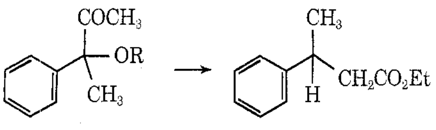
The first examples of α-hydroxy ketones undergoing the Favorskii reaction are presented. The alcohols 1-acetyl-1-hydroxy-5-methoxy-1,2,3,4-tetrahydronaphthalene (**1a**) and 3-phenyl-3-hydroxy-2-butanone (**2a**) undergo the rearrangement with NaH in diethyl carbonate to give the esters ethyl 5-methoxy-1,2,3,4-tetrahydronaphthalene-1-acetate (**3a**) and ethyl 3-phenylbutyrate (**4**), respectively. Possible mechanisms are discussed, and a cyclopropanone intermediate has been verified by the use of the $^{18}$O-labeled ketone (**2a**).

Apart from α-halo ketones,[2] there is only one reported instance[3] of an effective α leaving group (2,3-epoxy ketones) generating the necessary intermediate, under the right experimental conditions, to yield the "Favorskii" type of products. We now wish to report a demonstration that the α-hydroxy ketones **1a** and **2a** can undergo the Favorskii rearrangement under suitable conditions.



**1a**, R = H
**b**, R = Ac

**3a**, R = COOEt
**b**, R = COOH
**c**, R = $CH_2OH$



**2a**, R = H
**b**, R = $CH_3$

**4**

The α-hydroxy ketone **1a** was prepared by the mercury-catalyzed hydration of 1-ethynyl-1-hydroxy-5-methoxy-1,2,3,4-tetrahydronaphthalene, which in turn was obtained from 5-methoxy-1-tetralone and acetylenemonomagnesium bromide.[4] Treatment of **1a** with NaH in diethyl carbonate at 100° under $N_2$ gave (instead of the desired Claisen condensation) the ester **3a** in nearly quantitative yield. The structure **3a** was confirmed by hydrolysis to the corresponding acid **3b**, and also by its reduction to the alcohol **3c** (3,5-dinitrobenzoate). Under the same conditions, the analogous open-chain alcohol **2a** was converted to **4** and identified by comparison (tlc and ir) with an authentic sample.

It is generally believed that the Favorskii reaction of an α-halo ketone with an α′ hydrogen atom occurs *via* a symmetrical (cyclopropanone)[5] intermediate, whereas α-halo ketones devoid of an α′ hydrogen follow a semibenzilic mechanism.[6]

The cyclopropanone mechanism is feasible in the present instance and could operate if the tertiary alcohol **2a** were converted into a carbonate leaving group. This could occur (Scheme I) by base-catalyzed transesterification with diethyl carbonate to form a mixed carbonate ester, with loss of ethoxide ion. Proton abstraction from the α-methyl group and internal attack with displacement of carbonate ion would then give the cyclopropanone **5**. Attack by ethoxide ion and collapse of the intermediate **6** by cleavage of bond a to afford the rearranged ester **4** correlates well with the expected[7] greater stability of the benzylic carbanion **8** over the primary carbanion **9** formed by fission of bond b. No trace of the isomeric ester **7** was found.

The semibenzilic mechanism, proceeding *via* an intermediate dianion, which was originally proposed[6] only for α-halo ketones without an α′ hydrogen, has in fact been shown[8] to occur also in α-halo ketones with an

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