98° (mixture melting point with authentic[11] benzyl *n*-butyl sulfone, m.p. 94°, was 96–97°).

The reaction of VIII *and* IX *with phenyl lithium.* The apparatus employed was a 100-ml. three-necked flask, fitted with a condenser, nitrogen inlet, stirrer, and dropping funnel. The sulfide VIII and IX (5.04 g., 0.0211 mole) was added dropwise (15 min.) to a solution of phenyllithium, prepared[12] from bromobenzene (3.65 g., 0.0232 mole, and lithium, 0.34 g., 0.049 g.-atom) in ether (20 ml.). The mixture was stirred in a nitrogen atmosphere for 3 hr. at room temperature. The precipitated salts (containing the lithium salt of methyl and *n*-butyl mercaptans) were removed by filtration and discarded. The ether layer was acidified with dilute hydrochloric acid, washed with water, and dried over $MgSO_4$, and concentrated. The residual oil was distilled, and there was obtained a low boiling fraction (1.24 g.) and a high boiling fraction (1.60 g., b.p. 99–108°/0.06 mm., n_D^{25} 1.603). The higher boiling fraction was identified by physical properties, and by infrared spectra, as nearly pure starting sulfide (31.7% recovery).

The lower boiling fraction was a mixture containing phenylacetylene (infrared absorption at 3315 cm.$^{-1}$ and 2100 cm.$^{-1}$), non-terminal acetylene (infrared absorption at 2160 cm.$^{-1}$), and other products. An attempt to resolve this material by distillation was not successful since the quantities of each component present, other than phenylacetylene, was relatively small.

The amount of phenylacetylene present was estimated by its conversion into mercury phenylacetylide. The amount of derivative isolated (m.p. 125–126°, mixture m.p. 125–126°) corresponded to a 35.3% yield of phenylacetylene. This is a minimum quantity since some phenylacetylene was undoubtedly lost during the distillation.

MINNEAPOLIS 14, MINN.

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[11] J. Buchi, M. Prost, H. Eichenberger, and R. Liberherr, *Helv. Chim. Acta*, **35**, 1527 (1952).

[12] G. Wittig, *Newer Methods of Preparative Organic Chemistry*, Interscience Publishers, Inc., New York, N. Y., 1948, p. 576.

[CONTRIBUTION FROM THE SCHOOL OF CHEMISTRY OF THE UNIVERSITY OF MINNESOTA]

Formation of Carbenes from α-Haloesters[1]

WILLIAM E. PARHAM AND FREDERIC C. LOEW[2]

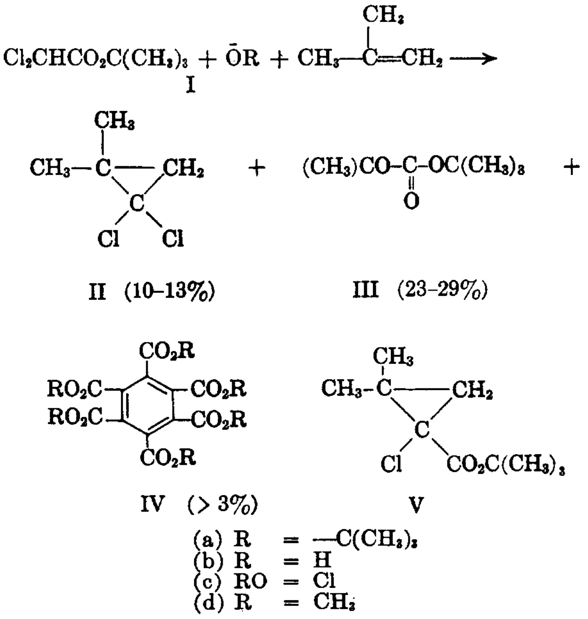
Received May 19, 1958

The reaction of butylate ion with the *t*-butyl esters of chloroacetic, dichloroacetic and trichloroacetic acid has been studied, and it has been shown that carbene intermediates result from the di- and tri-chloro esters. The reaction of *t*-butyl dichloroacetate with butylate ion, in the presence of isobutylene, results in the formation of 1,1-dichloro-2,2-dimethylcyclopropane (II), di-*t*-butyl carbonate (III) and hexa-*t*-butyl mellitate (IVa). The reaction of the trichloro ester, under similar conditions, affords di-*t*-butyl carbonate (III) and the cyclopropane II. Thus, under the conditions employed, both of these esters are converted into dichlorocarbene.

In a previous communication[3] we suggested that the reaction between esters of dichloroacetic acid and base might lead to carbenes. However, when indene was used as the carbene acceptor no products were obtained which could have been derived from chlorocarbalkoxy carbene. We have now made a study of the action of the *t*-butylate ion upon the *t*-butyl esters of chloroacetic, dichloroacetic, and trichloroacetic acid and have found that carbenes result from the last two.

The reaction of *t*-butyl dichloroacetate with *t*-butylate ion in the presence of isobutylene was first studied, and the products isolated were 1,1-dichloro-2,2-dimethylcyclopropane (II), *t*-butyl carbonate (III) and a neutral ester which has been shown to be hexa-*t*-butyl mellitate (IVa).

The isolation of 1,1-dichloro-2,2-dimethylcyclopropane (II) from the above reaction is convincing evidence for the formation of dichlorocarbene (VII) as an intermediate.[3-6] The formation of this car-



(a) R = —$C(CH_3)_3$
(b) R = H
(c) RO = Cl
(d) R = CH_3

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[1] This work was supported by a grant (NSF-G2163) from the National Science Foundation.

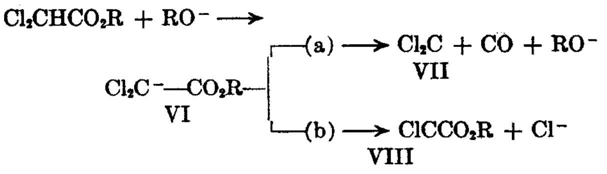
[2] Sinclair Refining Company Fellow, 1956–1957.

[3] W. E. Parham and R. R. Twelves, *J. Org. Chem.*, **22**, 730 (1957).

[4] (a) J. Hine, *J. Am. Chem. Soc.*, **72**, 2438 (1950); (b) J. Hine and A. M. Dowell, Jr., *J. Am. Chem. Soc.*, **76**, 2688 (1954); (c) J. Hine, P. C. Peek, Jr., and B. D. Oakes, *J. Am. Chem. Soc.*, **76**, 6162 (1954); (d) J. Hine, A. M. Dowell, Jr., and J. E. Singley, Jr., *J. Am. Chem. Soc.*, **78**, 479 (1956).

[5] (a) W. von E. Doering and A. K. Hoffmann, *J. Am. Chem. Soc.*, **76**, 6162 (1954); (b) W. von E. Doering and P. LaFlamme, *J. Am. Chem. Soc.*, **78**, 5448 (1956).

bene is rationalized by equation (a) below; carbon monoxide was identified[7] as a reaction product.[8]

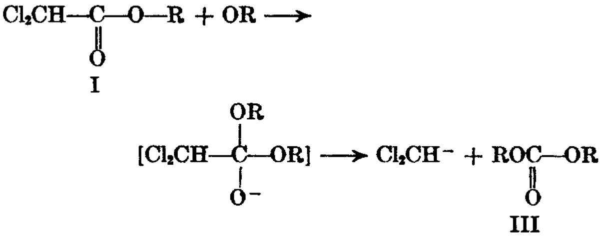


$$Cl_2CHCO_2R + RO^- \longrightarrow \underset{VI}{Cl_2C^-{-}CO_2R} \begin{cases} \xrightarrow{(a)} \underset{VII}{Cl_2C} + CO + RO^- \\ \xrightarrow{(b)} \underset{VIII}{ClCCO_2R} + Cl^- \end{cases}$$

It is interesting to note that the cyclopropyl ester V was not found as a reaction product; consequently it was concluded that either: (a) the carbene VIII was not formed (reaction b) in appreciable quantity, or (b) the carbene VIII does not add appreciably to isobutylene. The former explanation is preferred in view of the electrophilic nature of carbene centers[6b] and the probable relative instability[6a] of carbene VIII with respect to the carbanion VI and carbene VII.

The formation of hexa-*t*-butyl mellitate (IVa) was unexpected; the assignment of structure was made on the basis of the following data: (1) the neutral ester had the composition (C and H) and molecular weight commensurate with IVa, and the infrared spectrum showed carbonyl absorption at 1730 cm.$^{-1}$, (2) the ester was hydrolyzed by action of 20% hydrochloric acid to mellitic acid (IVb), and the latter was converted by conventional methods into the acid chloride (IVc), and methyl ester (IVd). The properties of IVc and IVd were in agreement with those previously reported for these derivatives, and the methyl ester (IVd) was shown to be identical with a sample of hexamethyl mellitate prepared from hexamethyl benzene. Whether hexa-*t*-butyl mellitate (IVa) was formed by trimerization of the possible intermediate di-*t*-butyl acetylenedicarboxylate,[9] or by some other mechanism is the subject of a study to be reported at a later date.

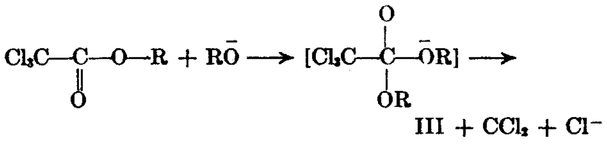
In view of the steric requirements imposed by the bulky *t*-butoxide groups, the formation of *t*-butyl carbonate (III), in relatively large amounts, was unexpected. The formation of this ester is thought to occur[10] as illustrated below. The fate of the Cl_2CH^- ion was not determined; however, there was no evidence for the formation of appreciable quantities of 1-chloro-2,2-dimethylcyclopropane, which could result if Cl_2CH^- lost chloride ion to give HCCl, or methylene chloride.



$$\underset{I}{Cl_2CH{-}\underset{\underset{O}{\|}}{C}{-}O{-}R} + OR \longrightarrow$$

$$[Cl_2CH{-}\overset{OR}{\underset{O^-}{C}}{-}OR] \longrightarrow Cl_2CH^- + \underset{III}{RO\underset{\underset{O}{\|}}{C}{-}OR}$$

The carbonate cleavage observed for I, suggested that α-haloesters which do not contain α-hydrogen atoms may serve as convenient sources for carbenes. This apparently is the case, since a 55% yield of pure 1,1-dichloro-2,2-dimethylcyclopropane (II), together with a 91% yield of di-*t*-butyl carbonate (III), resulted from the reaction of *t*-butyl trichloroacetate[11] and potassium *t*-butylate in the presence of isobutylene.



$$Cl_3C{-}\underset{\underset{O}{\|}}{C}{-}O{-}R + R\bar{O} \longrightarrow [Cl_3C{-}\overset{O}{\underset{OR}{C}}{-}\bar{O}R] \longrightarrow III + CCl_2 + Cl^-$$

The reaction of *t*-butyl chloroacetate and potassium *t*-butylate, in the presence of 2-methylbutene-2, was investigated. Tars resulted, and no *t*-butyl carbonate or volatile halogen containing products were noted.

EXPERIMENTAL

The reaction of t-butyl dichloroacetate (I) with potassium t-butylate in the presence of isobutylene. t-Butyl dichloroacetate[3] (185 g., 1.0 mole) was added over a 90 minute period to a cold (−6 to −10°) mixture of powdered potassium *t*-butoxide[5a] (1.28 mole), isobutylene (300 ml. at −80°, *ca.* 4 mole), and olefin-free pentane (300 ml.). The resulting deep red mixture was stirred for an additional 90 min., the dry ice condenser and cooling bath were removed, and the isobutylene was allowed to evaporate (overnight). Water (300 ml.) was added to the reaction mixture to dissolve solids, and the organic layer was separated and dried ($MgSO_4$). After several days, a solid precipitated from this solution. The mixture was filtered and processed as described below.

Hexa-t-butyl mellitate (IVa). The solid, containing magnesium sulfate, was treated with water, and the organic component was collected by filtration. This material was triturated with methanol and recrystallized from benzene-petroleum ether. There was obtained 3.2 g. (2.8% yield) of IVa (dec. upon heating, does not melt below 360°).

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(6) (a) P. S. Skell and A. Y. Garner, *J. Am. Chem. Soc.*, **78**, 3409 (1956); (b) P. S. Skell and A. Y. Garner, *J. Am. Chem. Soc.*, **78**, 5430 (1956); (c) P. S. Skell and R. C. Woodworth, *J. Am. Chem. Soc.*, **78**, 4496 (1956); (d) P. S. Skell and R. M. Etter, *Chem. and Ind.* (*London*), 624 (1958).

(7) E. G. Voiret and A. L. Bonaimé, *Ann. chim. anal.* 26, 11 (1944).

(8) Hine and coworkers [J. Hine, E. L. Pollitzer, and H. Wagner, *J. Am. Chem. Soc.*, **75**, 5607 (1953)] have found that alcohols are dehydrated in the presence of haloforms and alkali with the formation of CO. Therefore, the detection of CO as a product in this reaction is not positive evidence for reaction path a. However, the dehydration of alcohols under these conditions can best be explained by assuming the intermediate formation of dichlorocarbene. Thus, although the CO may be formed primarily by dehydration of *t*-butyl alcohol, reaction path a appears to be the only plausible route to dichlorocarbene.

(9) O. Diels, *Ber.*, **75B**, 1452–67 (1942) has shown that hexamethyl mellitate is formed from dimethyl acetylenedicarboxylate by the action of pyridine acetate.

(10) See M. L. Bender, *J. Am. Chem. Soc.*, **75**, 5986 (1953).

(11) This observation supports the suggestion that difluorocarbene may result from the reaction of ethyl trifluoroacetate and ethoxide ion [E. Bergman, *J. Org. Chem.*, **23**, 476 (1958)]. Bergman also reports that the reaction of higher perfluoroesters with alkoxide leads to the formation of diethyl carbonate.

Anal. Calcd. for $C_{36}H_{54}O_{12}$: C, 63.68; H, 8.02; mol. wt., 678. Found: C, 63.56; H, 7.76; mol. wt. (cryoscopic in benzene), 700.

The infrared spectrum in Nujol showed peaks at 2940 (s), 2860 (s), 1730 (s), 1465 (m), 1425 (w), 1395 (w), 1370 (m), 1335 (m), 1260 (s), 1238 (s), 1150 (s), 1113 (m), 1035 (w), 985 (m), 905 (w), 883 (m), 853 (m), 838 (m), 785 (w), 735 (m), 717 (w), 685 (w), and 658 cm.$^{-1}$ (m).

The organic solution, described above, was distilled through a 12 inch glass-helices packed column. After removal of pentane, four fractions were collected: (1) b.p. 50–55°/135 mm., consisting mainly of *t*-butyl alcohol, (2) 14.34 g., b.p. 60–63°/115 mm., n_D^{25} 1.4320–1.4430, (3) 39 g., b.p. 88°/65 mm., m.p. 35–40°, and (4) residue, dark in color, 59 g.

1,1-Dichloro-2,2-dimethylcyclopropane (II). Fraction 1 was dissolved in petroleum ether, washed with 5 portions of water, dried ($MgSO_4$), and distilled at atmospheric pressure. There was obtained, in addition to *t*-butyl alcohol, 8.2 g. of II, b.p. 118–120°, n_D^{25} 1.4410–1.4462. This material was combined with fraction 2, above, and redistilled through a Piros-Glover spinning band column. There was obtained 18.2 g. (13.1%) of II, b.p. 118–120°, n_D^{25} 1.4466 (reported[5a] b.p. 118–120°, n_D^{25} 1.4454).

Anal. Calcd. for $C_5H_8Cl_2$: C, 43.19; H, 5.80. Found: C, 43.38; H, 5.80.

The infrared spectrum of the product showed bands at 1735, 1290, and 1140 cm.$^{-1}$, attributed to small contamination by di-*t*-butyl carbonate (III). This impurity was easily removed by treating a pentane solution of the cyclopropane with several portions of concentrated sulfuric acid. The spectrum of the resulting material (n_D^{25} 1.4468) was identical with that of an authentic sample of II prepared, as previously described,[5a] from chloroform.

Di-t-butyl carbonate (III). Fraction 3 (m.p. 35–40°), described above, was principally di-*t*-butyl carbonate (III, 23–29% yield in several runs). The product was recrystallized from methanol, and the resulting product (m.p. 39.5–40.5°, yield range 16–20%) was identical (mixture m.p. and I.R. spectra) with an authentic sample of III.[12]

Infrared bands at 3000 cm.$^{-1}$ (s), 2960 (m), 1735 (v.s.), 1475 (w), 1460 (w), 1395 (m), 1370 (s), 1290 (v.s.), 1250 (w), 1140 (v.s.), 895 (w), 845 (m), 790 (w), and 715 (w). 5% in CCl_4—CS_2 using a 37 μ cell.

Residue. The residue (59 g.), described above, was chromatographed on a column of Alcoa alumina (600 g.), using petroleum ether and mixtures of petroleum ether and benzene, as eluant. A pale yellow resinous material (15 g.) was isolated. This material showed unsaturation (I.R. absorption at 1620 cm.$^{-1}$), ester carbonyl (I.R. absorption at 1725 cm.$^{-1}$, shoulder at 1740 cm.$^{-1}$), and chlorine (sodium fusion). The material decomposed upon attempted distillation (short path) at 0.1 mm. This product was not identified, but was considered to be higher condensation products of *t*-butyl dichloroacetate.

Attempts to isolate 1-chloro-2,2-dimethylcyclopropane in the above reaction were unsuccessful.

The reaction of *t*-butyl dichloroacetate, potassium *t*-butylate and isobutylene, was repeated in the absence of the solvent pentane. No evidence for the formation of either methylene chloride or 1-chloro-2,2-dimethylcyclopropane was noted.

Proof of structure of hexa-t-butyl mellitate (IVa). *Hydrolysis of* IVa *to mellitic acid* (IVb). A mixture of hexa-*t*-butyl mellitate (3.4 g.) and 20% hydrochloric acid (25 ml.) was heated at the reflux temperature until all the solid material had dissolved (5–6 hr.). The resulting solution was evaporated to dryness, and the residue (1.7 g.) was recrystallized three times from 20% HCl. The acid did not have a sharp melting point (285–299°, uncorrected). Mellitic acid is reported[13] to melt at 285–287°.

Anal. Calcd. for $C_{12}H_6O_{12}$: C, 42.11; H, 1.77; Neut. eq., 57.0. Found: C, 42.46; H, 2.00; Neut. eq., 57.0.

Conversion of mellitic acid to the acid chloride (IVc). The acid IVb (0.1 g.) was converted to the acid chloride (IVc) by reaction with phosphorous pentachloride (0.7 g.) at 150° for 2 hr. The resulting solid was recrystallized from benzene, and sublimed (flame) at 1 mm. The product sublimed at 240–245° (reported m.p. 240°).[14]

Anal. Calcd. for $C_{12}O_6Cl_6$: C, 31.84; H, 0.00. Found: C, 31.97; H, 0.24.

Conversion of mellitic acid (IVb) *to hexamethyl mellitate* (IVd). Mellitic acid (0.9 g.) was esterified by reaction with excess diazomethane in ether. The crude product (1.3 g.) was recrystallized from benzene-petroleum ether which afforded 1.15 g. of colorless hair-like needles, m.p. 192–193°. Analysis of this material gave values consistent with the formula $C_{18}H_{18}O_{12}\cdot{}^1/_3\ C_6H_6$. The sample was redried (100°/0.1 mm. for 3 hr.) and the resulting product melted at 188–188.5° (reported[15] m.p. 187–188°).

Anal. Calcd. for $C_{18}H_{18}O_{12}$: C, 50.72; H, 4.23; mol. wt. 426. Found: C, 50.98; H, 4.08; mol. wt. (cryoscopic in benzene), 430, 437.

This material proved identical (m.p., mixture m.p., infrared spectrum) with authentic IVd.

Authentic hexamethyl mellitate (IVd). Mellitic acid was prepared[13] by oxidation of hexamethyl benzene (2 g.) with a mixture of fuming nitric acid (7 ml., d. 1.52) and water (2 ml.). The Carius tube employed had a volume of 100 ml. and a wall thickness of 3 mm. Attempts to carry out this reaction on a larger scale, or in smaller tubes, resulted in failure of the tube. Recrystallization of the product from 65% HNO_3 afforded 1.18 g. (28%) of mellitic acid.

This material was methylated with diazomethane as described above. The product was recrystallized twice from benzene-petroleum ether B and dried at 100° (0.1 mm.), m.p. 187–189° (reported[15] 187–188°).

Authentic 1,1-dichloro-2,2-dimethylcyclopropane (II). This compound was prepared from potassium *t*-butylate (0.40 mole), isobutylene (160 ml. at −80°), and chloroform (90 g.) according to the procedure of Doering and Hoffmann,[5a] except that pentane (150 ml.) was used as the solvent rather than *t*-butyl alcohol (obviating extensive washings of the reaction mixture and foreruns). The reaction mixture was washed with two portions of water, separated, dried ($MgSO_4$), and distilled to afford 33.5 g. (60%) of product, b.p. 118–20/738 mm., n_D^{25} 1.4468.

Authentic di-t-butyl carbonate. The procedure of Choppin and Rogers[12] was used except that potassium *t*-butylate in hexane was substituted for sodium *t*-butylate in dioxane-*t*-butyl alcohol. Phosgene (13.8 g., 0.139 mole) was bubbled into the warm (60°) suspension of potassium *t*-butylate (0.255 mole) in hexane (200 ml.) in a stream of nitrogen. After the addition was completed (1 hr.), the reaction mixture was poured into 200 ml. of ice and water. The organic layer was separated, dried ($MgSO_4$), and distilled. The distillate (16.7 g. (75%), b.p. 88/65 mm., m.p. 33–38) was recrystallized twice from methanol-water to yield 9.8 g. (44%) of di-*t*-butyl carbonate, m.p. 39.5–40.5° (reported m.p., 39.5–40.5°).

The reaction of t-butyl trichloroacetate with potassium t-butoxide in the presence of isobutylene. The reaction of *t*-butyl trichloro acetate[16] (205 g., 0.94 mole), potassium *t*-butylate (1.25 mole), and isobutylene (300 ml. at −80°, *ca.* 4 moles)

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[12] A. R. Choppin and J. W. Rogers, *J. Am. Chem. Soc.*, **70**, 2967 (1948).

[13] J. P. Wibaut, J. Overhoff, E. W. Jonker, and K. Gratama, *Rec. trav. chim.* **60**, 742 (1941).

[14] A. Claus, *Ber.*, **10**, 561 (1877).

[15] H. v. Pechmann, *Ber.* **31**, 502 (1898).

[16] W. E. Scovill, R. E. Burk, and H. P. Lankelma, *J. Am. Chem. Soc.*, **66**, 1039 (1944).

in pentane (300 ml.) solvent was carried out according to the procedure described for the dichloro ester. The final organic solution was distilled. The material boiling at 62–67°/115 mm. (92 g., n_D^{25} 1.4372) was redistilled to give 72 g. (b.p. 67°/115 mm., n_D^{25} 1.4468, 55.5% yield) of pure 1,1-dichloro-2,2-dimethylcyclopropane (II). The yield of di-*t*-butyl carbonate (148 g., b.p. 89°/65–70 mm., m.p. 38–40°) was 91%.

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[CONTRIBUTION FROM THE NOYES CHEMICAL LABORATORY, UNIVERSITY OF ILLINOIS]

Synthesis of Medium- and Large-Ring Ketones *via* the Dieckmann Condensation

NELSON J. LEONARD AND C. W. SCHIMELPFENIG, JR.

Received May 19, 1958

The Dieckmann cyclization of a series of α,ω-diesters, diethyl suberate through diethyl thapsate, has been carried out with potassium *t*-butoxide in xylene under high dilution conditions with high speed stirring in a nitrogen atmosphere, and some medium- and large-ring monoketones and large-ring diketones, with carbonyl groups diametric, have been obtained. The relative yields have been compared with those of the corresponding carbocyclic ketones and diketones obtained by other methods.

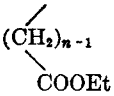
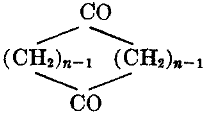
The concept has been generally held that the Dieckmann cyclization of diesters is applicable only to the formation of the classical rings of five, six, and seven members, as originally employed.[1] With the conviction that suitable conditions could extend the usefulness of this reaction and in the interest of providing an additional route to larger cyclic ketones, we have examined the relative proportions of monoketones and diketones obtainable following the cyclization of a series of α,ω-diesters with potassium *t*-butoxide in xylene under high dilution conditions in a nitrogen atmosphere.

These cyclization conditions were employed originally for the synthesis of an eight-membered ring aminoketone by Sentz[2] in this laboratory, who also suggested their application to the formation of cycloakanones of greater ring size than seven members. Potassium *t*-butoxide has been used successfully for the cyclization of various aminodiesters to eight-[3–7] and ten-membered[6] ring aminoketones and sixteen-[6] and twenty-membered[6] ring diaminodiketones. Sodium hydride, which Blicke and his co-workers[8] employed for the synthesis of cycloöctanone *via* a Dieckmann condensation, has also been used for the preparation of eight-membered[6,9,10] ring aminoketones and sixteen-membered[9] ring diaminodiketones.

In the present investigation we have carried out the Dieckmann cyclization of the esters (I), diethyl suberate through diethyl thapsate, under identical conditions, employing potassium *t*-butoxide in xylene, under high dilution conditions with high speed stirring in a nitrogen atmosphere. A uniform isolation procedure was used and the monoketones (II) and diketones (III) were collected and identified, while no attempt was made to characterize any triketones or polymeric products. The yields of mono- and diketones are given in Table I. It will be observed that the conditions employed are useful for obtaining the cyclic 14- and 15-membered monoketones and the 18-, 20-, 22-, and

TABLE I

YIELDS OF KETONES PRODUCED BY THE DIECKMANN CYCLIZATION

	Actual Yield, %	
Diester (I)	Monoketone (II)	Diketone (III)
	$(CH_2)_{n-1}CO$	
$n =$ 7	47	0
8	15	11
9	0	28
10	0	12
11	0.53	23
12	0.47	16
13	24	19
14	32	2.2
15	48	0.94

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(1) (a) W. Dieckmann, *Ber.*, **27**, 102 (1894); (b) W. Dieckmann, *Ann.*, **317**, 27 (1901).

(2) R. C. Sentz, Ph.D. thesis, University of Illinois, 1952.

(3) N. J. Leonard and R. C. Sentz, *J. Am. Chem. Soc.*, **74**, 1704 (1952).

(4) N. J. Leonard, S. Swann, Jr., and E. H. Mottus, *J. Am. Chem. Soc.*, **74**, 6251 (1952).

(5) N. J. Leonard, S. Swann, Jr., and Glenn Fuller, *J. Am. Chem. Soc.*, **76**, 3193 (1954).

(6) N. J. Leonard, M. Ōki, and S. Chiavarelli, *J. Am. Chem. Soc.*, **77**, 6234 (1955).

(7) N. J. Leonard, D. F. Morrow, and M. T. Rogers, *J. Am. Chem. Soc.*, **79**, 5476 (1957).

(8) F. F. Blicke, J. Azuara, N. J. Doorenbos, and E. B. Hotelling, *J. Am. Chem. Soc.*, **75**, 5418 (1953).

(9) N. J. Leonard and M. Ōki, *J. Am. Chem. Soc.*, **77**, 6241 (1955).

(10) N. J. Leonard and M. Ōki, *J. Am. Chem. Soc.*, **77**, 6245 (1955).